Forensic Investigation of Clandestine Laboratories

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Introduction

Drugs of abuse in the United States have traditionally come from a variety of foreign sources. Heroin and cocaine are produced in foreign countries. A vast amount of marijuana is cultivated and smuggled in from sources outside the United States. However, law enforcement authorities must look inward to identify the source of clandestinely produced synthetic drugs that are increasing in popularity.

Clandestinely produced drugs of abuse are not the only controlled substances affecting the public order. The clandestine production of explosives and explosive mixtures placed into destructive devices and used with criminal intent has greatly impacted the feelings of safety experienced by law-abiding citizens.

The manufacturing of controlled substances in clandestine labs is an ever-increasing problem within the United States. Identifying and shutting down these operations has the greatest impact in stemming the flow of contraband substances. The effect of eliminating the ultimate source of the controlled substance being manufactured reaches far beyond jailing individuals arrested at the site. Everyone who would potentially have come in contact with the finished product, from the mid-level distributors to the end users, feels the ramifications of putting the manufacturer out of business.

The investigation of clandestine labs is one of the most challenging of law enforcement. It is a roller-coaster ride of activity that requires every tool at its disposal. Traditional investigative techniques are used to develop information concerning the location of the clandestine lab and the identity of the operator. Forensic experts are used to corroborate information by establishing the identity of the final products as well as the manufacturing methods used to produce them.

No other law enforcement activity relies on forensic experts as heavily as does the investigation of clandestine labs. The forensic expert's involvement commences with the drafting of the affidavit used to obtain the search warrant. His or her expertise is imperative to effectively process the crime scene. Experts analyze the samples from the crime scene in a forensic laboratory. Finally, they render opinions in a written report or in courtroom testimony. Occasionally, the forensic expert may be called upon further to testify on

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auxiliary issues concerning the clandestine lab investigation that occur even after the criminal case has been adjudicated.

A team effort is necessary for identifying, investigating, and prosecuting a clandestine lab. It is a collaboration of the efforts of law enforcement, forensic experts, scientists, and criminal prosecutors to present a case that definitively demonstrates how a group of items with legitimate uses is being used to manufacture an illegal controlled substance. *Forensic Investigation of Clandestine Laboratories* was written to provide these groups with the general information needed to understand how the different pieces of the clandestine lab puzzle fit together.

Individuals outside of law enforcement can benefit from the information in the first three chapters of this book. Emergency responders, such as police patrol officers, firefighters, emergency medical technicians (EMTs), and representatives from certain social service agencies, routinely encounter clandestine labs. Landlords, storage locker managers, and the public at large stumble upon these operations without realizing it. The knowledge gained from reading this text will allow these groups to be able to recognize a potentially dangerous situation so they can report it to the appropriate authorities.

The goal is to provide anyone involved in the investigation or prosecution of clandestine lab activity the information to guide him or her through the process of establishing the existence of a clandestine lab beyond a reasonable doubt. Just by reading this book, the reader will not be an expert in the clandestine manufacture of controlled substances. That can only be accomplished through training and experience.

The information in this book will provide an overview of clandestine labs. This will be accomplished by dividing the process into five sections that correspond to the various phases of investigation and prosecution. Described in the first section is how to recognize clandestine labs and the physical characteristics they have in common. In the second section, processing the site of a clandestine lab will be reviewed. Covered in the third section are the analytical techniques that can be used in the laboratory to analyze evidence from a clandestine laboratory. Presented in the fourth section are the opinions that can be rendered from the physical evidence. In the fifth and final section, presenting the evidence in court is covered.

Recognition of clandestine lab activity is the first step in the process. In Chapters 1 through 3, a clandestine lab is described, along with the common elements to expect. A profile of a clandestine lab operator will be presented. And, chemical and equipment requirements, as well as the basic manufacturing techniques utilized, will be identified. In this section, the commonly held, yet faulty, notion that the manufacture of controlled substances requires higher education, sophisticated equipment, and exotic chemicals will be dispelled. The knowledge gleaned from this section should enable an individual

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to recognize a clandestine lab. An investigator should be able to articulate why a clandestine lab exists and, subsequently, secure a search warrant to proceed to the next phase of the process.

Some of the explanations of the manufacturing process may seem oversimplified to a forensic chemist. Yet investigators, attorneys, and jurors involved in the various segments of the investigation and prosecution of clandestine labs cannot be expected to have acquired the scientific knowledge necessary to understand the chemical processes involved. Using nontechnical terms, with common examples, should remove the scientific mystique. For a broad audience, the understanding of the process of clandestinely manufacturing a controlled substance is more easily achieved using laymen's terms.

In *Forensic Investigation of Clandestine Laboratories*, how to clandestinely make controlled substances is not described in detail. Unfortunately, there are already numerous sources of such information available to the general public. What is addressed in this book is generally how controlled substances are made; how investigators, forensic experts, scientists, and attorneys can identify the existence of a clandestine lab and compile the information necessary to establish what was being manufactured, and how it was being manufactured; and finally, how to present the information to a jury for adjudication.

Knowing what a clandestine lab is and proving one exists are separate issues. In Chapters 4 and 5, the steps necessary to collect and identify all of the pieces of the clandestine lab puzzle are presented. The information gathered from investigators must be evaluated. The steps required to process clandestine lab sites for physical evidence are outlined, and analytical approaches that can be taken during the subsequent laboratory analysis are described.

Processing the clandestine lab scene is addressed in Chapter 4. It is more complicated than the traditional crime scene search normally associated with a narcotics investigation. Because of the chemicals involved, the site of a clandestine lab is, by definition, a "hazardous materials incident" and necessitates invoking different protocols for crime scene processing. Agencies such as the fire department, emergency medical personnel, and local health and environmental quality personnel should be involved. The equipment requirements for processing clandestine lab scenes are more extensive because of the potential chemical exposures. Finally, there are a number of preliminary opinions that should be made when evaluating the physical evidence observed at the scene, which necessitates an on-scene expert.

Addressed in Chapter 5 are the options available to the forensic chemist who analyzes the evidentiary samples. Complete forensic laboratory analysis is a critical element of a clandestine lab investigation. The analysis of a reaction mixture is more complex than identifying the controlled substance

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it contains. Identification of precursor and reagent chemicals as well as reaction by-products is necessary to establish the manufacturing method used. Identification of unique chemical components can be used as an investigative tool to connect the clandestine lab under investigation to other illegal activity.

Opinions, or "What does it all mean?", are presented in the next section. A large amount of information is collected during a clandestine lab investigation. Dealt with in this section, is collating information from various sources and creating a profile of the clandestine lab under investigation. What type of operation existed? What was it making? How was it being made? How much could it make? These are some of the questions that will be addressed in Chapter 6.

All of the work to this point may be useless if the expert's opinions cannot be relayed effectively to a jury. Expert testimony is presented in the final section of the book. Discussed in Chapter 7 is how to effectively educate the prosecutor, deal with defense attorneys, and present technical information to nontechnical jurors.

The main focus of clandestine lab investigations in the United States is the manufacture of illicit drugs because the manufacturing of explosives is not illegal, per se. However, placing the explosive final product of a clandestine lab into a destructive device is illegal. All of the techniques used to investigate clandestine drug labs can also be applied to the manufacture of explosive chemicals, compounds, and mixtures. Issues involving the clandestine manufacture of explosives are addressed in Chapter 8.

The use of forensic evidence is essential to the successful investigation and prosecution of a clandestine lab, whether the final product is a drug or an explosive. The proper collection and preservation of the physical evidence followed by the complete analysis of the evidentiary samples are key elements. The information gathered is the cornerstone on which the forensic expert's opinion is based. If forensic evidence is properly handled, the Court will have all of the information it needs to make a fully informed decision.

Donnell R. Christian, Jr.

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1 Basic Clandestine Drug Manufacture

Before a clandestine lab investigation can begin, the investigator must be able to recognize that such a lab exists. To do this, he must be familiar with the basic techniques used to produce controlled substances. Provided in this chapter is information concerning basic manufacturing techniques used by clandestine lab operators. Legitimate sources of chemicals and equipment will be used in the explanations, followed by examples of underground alternatives.

Clandestine labs come in a variety of shapes and sizes; their sophistication is limited only by the education and imagination of the operator. Complicated equipment and exotic chemicals are not required to manufacture drugs of abuse or explosives (controlled substances). Most of the equipment and chemicals found in a lab have legitimate uses and can be obtained from a variety of legitimate retail outlets. Therefore, the forensic clandestine lab investigator must be able to recognize the combinations of equipment and chemicals that are used to manufacture controlled substances and to determine whether the combination is coincidental or intentional.

A clandestine laboratory is literally a secret room or building equipped for scientific research or manufacture. Clandestine labs may not be illegal. The substances they produce and the act of manufacturing them is what may be controlled.

Webster's Dictionary defines manufacturing as "…the act of making goods, by hand or machinery." The legal definition of manufacturing is different. The Code of Federal Regulations (21 CFR 1300.01) defines manufacture as "… the producing, preparing, propagating, compounding or processing of a drug or other substance or the packaging or repackaging of such substance or labeling or relabeling of the commercial container of such…" This definition relates more directly to the production of drugs of abuse and

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is applicable in one form or another throughout the United States. Investigators should compare the federal definition with their local statutes and consult with district attorneys responsible for prosecuting clandestine labs about any differences.

The legal definition includes acts of packaging and labeling that are not normally associated with the "making of goods." The acts of preparing the final product for distribution or sale expand the perception of laboratory operations from the traditional mixing and extracting of chemicals. Under this definition, the mirror act of making little ones out of big ones is the same as making the big ones to begin with.

Many criminal statutes dealing with the manufacture of controlled substances state that the possession of chemicals and equipment for the purpose of manufacturing a controlled substance is illegal. The statute may not specify that all of the equipment and chemicals must be present, only that a combination exist that is sufficient for a reasonable person to believe that a manufacturing operation exists. Other statutes may require all of the components of the operation to be present. Either situation places the burden of demonstrating how the different components can be utilized to manufacture a controlled substance on the government. Understanding the different manufacturing processes will allow the investigator or prosecutor to differentiate and articulate how the presence of cold medications, rubbing alcohol, coffee filters, and glass jars can be legitimately present in one situation and yet be used in a clandestine lab that manufactures a controlled substance in another.

The manufacture of explosives is a slightly different issue. The simple possession of explosives is not regulated to the extent drugs are. Explosives have a wide range of legitimate applications. However, for public safety reasons, the manufacture, distribution, and storage of explosives is regulated. In the interest of public safety, the U.S. government regulates the importation, manufacture, distribution, and storage of explosive material through 27 CFR 55 and Chapter 40 of Title 18 of the United States Code (18 USC 40). As with the drug laws, there may be additional restrictions on the possession, manufacture, distribution, and storage of explosives or explosive material that are enacted by state and local statutes.

1.1 Lab Operators

There are three distinct categories of clandestine lab operators: small-scale, commercial, and educated (Table 1.1). The size of the lab may vary among operator categories, but the principles that demonstrate an operation exists remain the same.

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Table 1.1 Operator Characteristics

Characteristic	Small-Scale Operator	Commercial Operator	Educated Operator
Chemical education	No	Yes	Yes
Drug user	Yes	Maybe	Maybe
For profit	No	Yes	Maybe
Legitimate chemical supply	No	Maybe	Yes
Single location operation	Yes	No	Yes
Local distribution	Yes	No	Maybe

The lab of a *small-scale operator* is the one most commonly encountered. He is generally a drug user, using his own product as well as selling a portion to support his habit. Financial gain is, therefore, often not the only objective of this operation. All stages of such an operation usually take place at one location. Generally, these operators have no chemical education. They obtain their chemicals through retail purchases at grocery stores and drugstores, local chemical supply houses, or mail order suppliers. At times, they will shoplift over-the-counter preparations that contain regulated precursor chemicals. Operations are conducted in single-family homes and apartments as well as hotel and motel rooms. These operations are found in the poorest part of a city or the most affluent. The types of motels and hotels used vary from skid row to major luxury chains. The distribution of the final product is usually in the same area as the lab, and the operator is usually the dealer.

The *commercial operator* manufactures for financial gain. These operators may or may not be users. Different portions of a commercial operation may take place at separate locations in an effort to avoid detection by law enforcement. The commercial operation may have one "cook" who holds the knowledge concerning the manufacturing process. He may or may not have chemistry training. The cook is usually only at the lab site during critical portions of the operation. For the balance of time, so-called "lab rats" are present to monitor the operation and secure it from theft or detection. The commercial operator has an established network to obtain large quantities of the necessary chemicals and equipment required to manufacture his product. A separate distribution network for the final product is usually established away from the lab to avoid detection. The commercial operator does not generally participate in street-level sales.

During the 1960s, outlaw motorcycle gangs began producing their own methamphetamine in these labs, and they dominated the distribution of the drug within the United States. Today, there are two major forces fueling the methamphetamine trade within the United States: well-organized manufacturing and trafficking groups based in Mexico, and a widely scattered series of local methamphetamine producers, predominantly based in rural areas

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around the country. They operate the well-organized, high-volume "superlab" defined by the Drug Enforcement Administration (DEA) as a clandestine lab operation that is capable of producing 10 pounds or more of methamphetamine in a single production cycle. The DEA estimates that less than 5% of clandestine labs seized are classified as superlabs. Concentrated in California and Mexico, they are estimated to produce over 80% of the methamphetamine available in the United States today.

The *educated operator* is the least-encountered type of clandestine lab operator. He usually has formal training in chemistry that was obtained through traditional education or from on-the-job training. He also has legitimate access to sources of regulated chemicals. He may even be using his job site as a manufacturing location without the knowledge of his employer. The educated operator may be a hybrid between the small-scale and the commercial operator. In some instances, he acts as the cook in operations that require chemical expertise, such as the synthesis of lysergic acid diethylamide (LSD) or fentanyl and its analogs. At other times, he can be found in a small-scale operation working as manufacturer and distributor. Graduate students, engineers, and government chemists have all been arrested for manufacturing controlled substances while using their employer's facility, or for purchasing chemicals and equipment through their employer without his knowledge. The educated operator may or may not have a distribution network established. He may or may not be a drug user. Profit is much more the driving force for the educated operator than for his uneducated small-scale counterpart.

1.2 Manufacturing Processes

There are a number of different processes that can be used to manufacture a controlled substance. The one employed will depend on the starting materials used and the end product desired. Each process may be encountered alone or in combination with one or more of the others. One clandestine lab may incorporate multiple manufacturing processes to obtain the end product. The four basic manufacturing processes used in clandestine labs are extraction, conversion, synthesis, and tableting.

The following is a generic example of a multimethod process. The necessary precursor chemical is extracted from a bulk substance and then converted into substance "B." Substance B is then combined with three other chemicals to synthesize compound "C." Compound C is extracted from the reaction mixture and then converted from the freebase into its salt form. The salt form is then extracted from the liquid, with the resulting product pack-

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aged (tableted) for distribution. This single operation, thus, actually contains seven processing steps.

It is not uncommon for large-scale clandestine lab operators to perform individual manufacturing processes at separate locations. This is once again done in an effort to avoid detection. Precursor chemical extraction may take place at Location 1. Synthesis and purification extractions then occur at Location 2. Conversion into the salt form may be done at a third location. And, packaging for sale may be done at the point of distribution.

The presence of only one process of the sequence does not make it any less a clandestine lab. It is the investigator's responsibility to recognize the process or part thereof and to articulate how it fits into the manufacturing method the operators are using.

1.2.1 Extraction Process

Extraction labs remove raw materials from a mixture. This is accomplished by using the desired component's physical and chemical properties to separate it from the mixture (Figure 1.1). No chemical change in the raw material occurs during the process. Examples of extraction labs include hashish production, coca paste productions, and extractions from pharmaceutical preparations.

Separating the resin containing the cannabinoids and THC from the marijuana leaves produces hashish and hash oil. Hashish is made by physically removing the resin from the leaves. The resin obtained is then collected and compressed into brick form. Hash oil is obtained by removing the resin from the leaves through the use of solvent extraction. In either case, the chemical structure of the extracted cannabinoids remains intact.

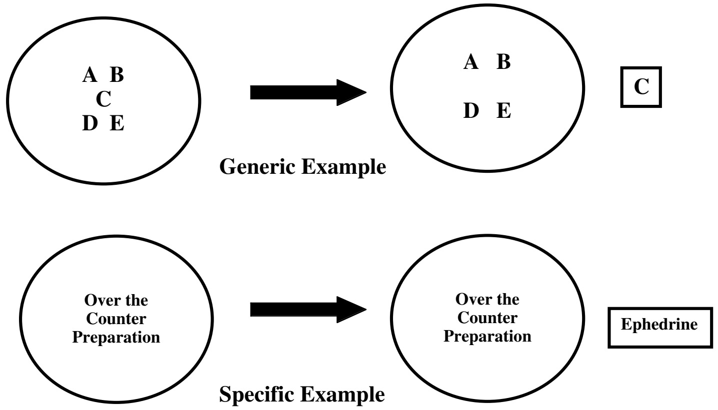

Figure 1.1 Extraction lab chemistry.

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The production of heroin begins with the removal of raw opium from the poppy. This physical extraction does not change the chemical structure of the morphine or codeine contained in the opium. It only removes it from the plant so the morphine can be processed into heroin. Some operations take the additional step of chemically extracting the morphine from the opium prior to conversion into heroin.

A number of over-the-counter pharmaceutical preparations contain the precursor chemicals used for the manufacture of controlled substances. These preparations are placed into a solvent, and the desired component is allowed to dissolve into the liquid. The liquid containing the component is removed from the solids and evaporated, leaving the component of interest.

Some pharmaceutical preparations contain a controlled substance in an aqueous solution. The item of interest is extracted from the solution by simply evaporating the water. This process may not be illegal. However, the resulting product may be. The act of extracting chemicals that are regulated from uncontrolled preparation may demonstrate the intent to do an illegal act.

1.2.2 Conversion Process

The conversion process takes a raw material and changes it into the desired product. This involves minor structural changes within the molecule of the compound, or of the chemical's *salt form*. Functional groups may be added or removed from the molecule, somewhat like pieces on a Tinkertoy®. The drug of interest can be changed from its salt form to the freebase form or from the freebase form to the salt form. Examples of the conversion process include the conversion of cocaine hydrochloride into freebase or "crack" cocaine and the conversion of ephedrine or pseudoephedrine into methamphetamine.

The simplest conversion process is the conversion of a freebase drug to its salt form or conversion from the salt form to the freebase (see Figure 1.2a). The simple act of adding a strong acid or a base to a water solution containing a drug will convert it to its salt or freebase form. This act changes the physical and chemical properties of the drug, allowing it to be extracted from liquid solutions. Depending on whether an acid or a base was added to the water, the drug either dissolves into or precipitates out of the water. Structurally, the drug remains unchanged.

The addition or removal of a functional group from a molecule is another form of a conversion process. A chemical reaction adds to, or takes away, a portion of the original compound, leaving the skeleton of the compound unchanged (see Figure 1.2b). The resulting molecule will have different physical and chemical properties. In the case of drugs, the original and final compounds will have different physiological effects on the body from each other.

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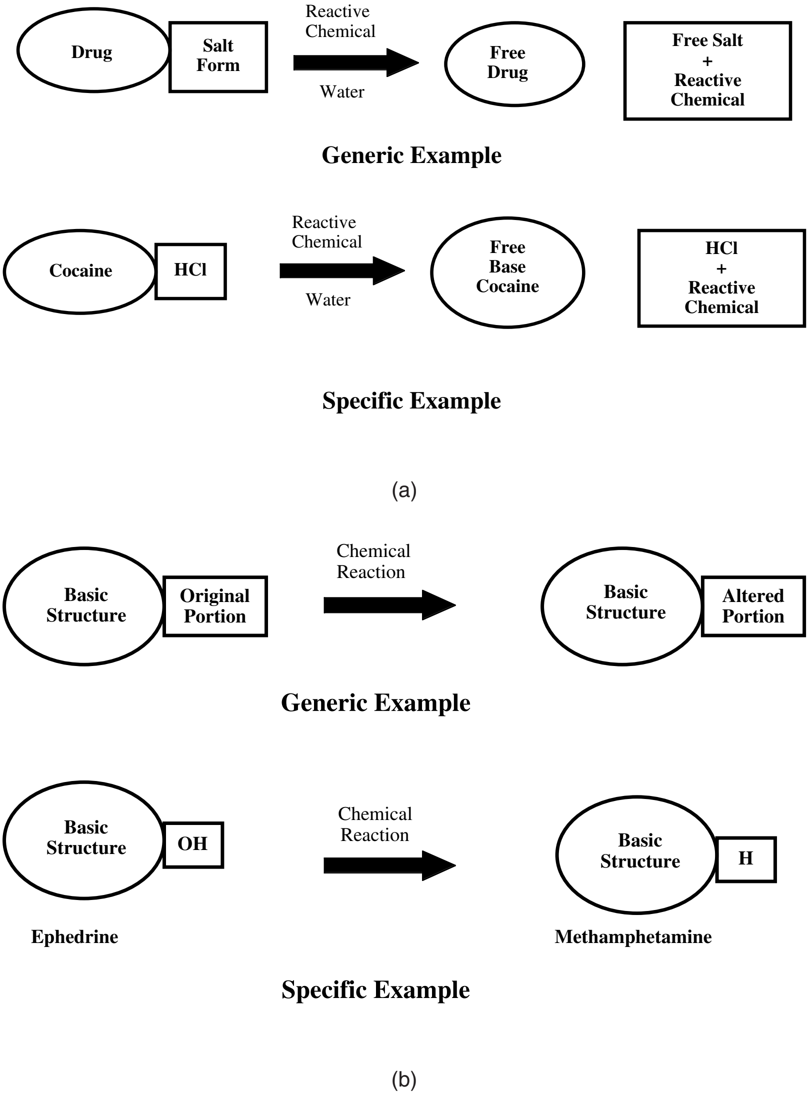

Figure 1.2 Conversion lab chemistry. (a) Salt form conversion. (b) Functional group conversion.

1.2.3 Synthesis Process

The synthesis process is a chemical reaction or series of chemical reactions in which molecules or parts of molecules are combined to create a new molecule. This process can be equated to a chemical erector set. It differs from the conversion process in that the skeleton of the resulting molecule is a sum of the molecules or significant parts of the molecules involved in the reaction. Lysergic acid diethylamide (LSD), phencyclidine (PCP), phenylac-

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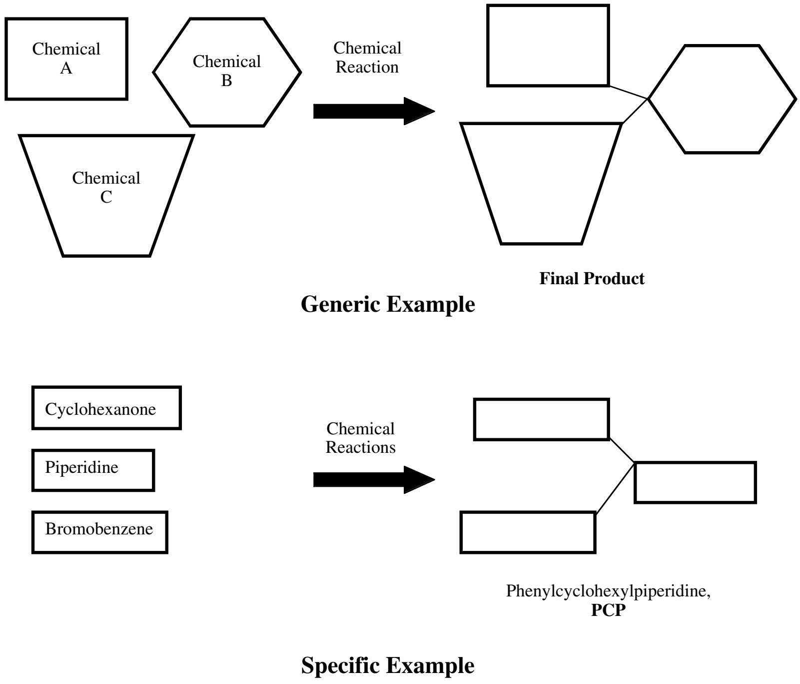

Figure 1.3 Synthesis lab chemistry.

etone (P2P), and certain methamphetamine reactions are examples of drugs produced using the synthesis process (Figure 1.3).

Even though the synthesis process sounds complicated, it may not require exotic equipment or lengthy reaction times. Some reactions can take place in plastic buckets in a matter of minutes or hours. Other reactions require sophisticated equipment or extended reaction times to achieve the desired results. The only way to differentiate between a conversion process and a synthesis process is to compare the structures of the precursor chemicals to those of the final product.

1.2.4 Tableting

Clandestine labs involved in the tableting process are placing the finished product into dosage forms or into smaller, more salable units for distribution. The tableting process derives its name from operations that place controlled substances into tablet form. The tableting process often includes pressing corporate logos onto the tablets to simulate legitimate pharmaceuticals.

Statutes in some jurisdictions include the act of packaging and repackaging in the definition of manufacturing. This language may interpret the act of placing the final product into a container for distribution (packaging)

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or dividing the container of final product into smaller containers (repackaging) as a manufacturing process. Thus, the act of making little ones out of big ones could also legally be equated to the actual production of the material.

This is a very subjective area. It is essential that the investigator consult with the local prosecutor to ascertain that the act of packaging items for sale or distribution can be statutorily considered manufacturing in that jurisdiction.

1.2.5 Combination Labs

A combination of processes is used to manufacture a controlled substance. It is not uncommon for more than one process to be observed at any given clandestine lab site. The size and scope of the operation will often determine how many processes are seen at the site.

A small-scale operator may extract the precursors needed from over-the-counter medication. He then converts the precursors into the controlled substance. Finally, he packages (tablets) the final product into dosage units or into smaller quantities for sale or distribution. All this can occur in a single hotel room, camping trailer, kitchen, or garage.

Commercial operators, in an effort to avoid detection, may choose to perform different phases of their operation at separate locations. The extraction of precursor chemicals from legitimate sources may occur at one location, and the synthesis may be done at another. The oily freebase compound is often transported to a third location and then converted to the solid salt form. The powder may then be transported to a final location, where it is prepared for distribution.

In both scenarios, the same processes existed: precursor chemicals were extracted; the precursor was converted or synthesized into the controlled substance; the controlled substance was converted into a usable form; and finally, the final product was packaged for distribution. The only difference is that in the first scenario, everything took place at the same location, while in the second, these processes occurred in four separate locations but under the same umbrella.

1.3 The Needs Triangle

Clandestine labs need equipment, chemicals, and knowledge to be complete. This Needs Triangle theme (Figure 1.4) is recurrent in many areas of science and life. As in any triangle, if any one of the three elements is eliminated, the system will not be complete. The amount of each component may vary, but each must be present for the operation to exist.

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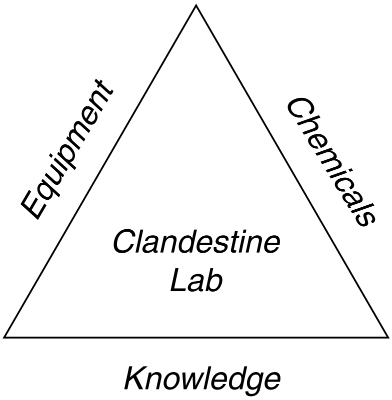

Figure 1.4 Clandestine lab needs triangle.

1.3.1 Equipment Needs

The equipment needs of a clandestine lab vary with the manufacturing method employed. Independent of the manufacturing method, two items are universally encountered: a triple-beam balance (found in any high school science lab), and surprisingly, pornography. Research indicates that methamphetamine and related psychomotor stimulants can increase the libido in users; this is in contrast to opiates, which decrease the libido. However, long-term methamphetamine use may be associated with decreased sexual functioning, at least in men. Pornographic materials and a triple-beam balance are seen so often in clandestine lab seizures that the inside joke of law enforcement is that their presence "officially" designates a clandestine lab.

All joking aside, common threads among the equipment needs of clandestine labs exist. Each manufacturing method will, of course, have its own equipment requirements that can usually be satisfied by using the scientific equipment that was designed to perform the process. However, to avoid detection, many clandestine lab operators designed alternatives to the traditional equipment, opting to use items that can be obtained from the grocery store or hardware store.

The manufacturing methods used by clandestine lab operators include reflux, distillation, and extractions. Understanding the mechanics of the scientific equipment used in the various manufacturing processes will allow the investigator to recognize the alternative equipment frequently encountered in clandestine labs.

1.3.1.1 Reflux

Refluxing is one of the most common methods used in the synthesis and conversion processes. This is a controlled boiling process in which the evap-

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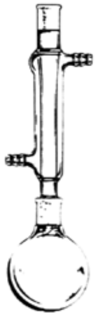

Figure 1.5 Basic reflux apparatus.

orated liquid is condensed and then returned to the reaction mixture. A slang term for the reaction mixtures found in clandestine labs is "soup," with the lab operator called the "cook." These are appropriate terms. In essence, the manufacturing of a controlled substance using the reflux method is similar to cooking soup.

When making soup, the cook combines the ingredients and boils them for a period of time. The only equipment necessary is a stove and a pot with a lid. When making a controlled substance using the reflux method, the cook combines the ingredients and refluxes them for a period of time. The refluxing apparatus is a specialized stove and a pot with a lid (Figure 1.5).

A source of heat is needed to cook the soup. A heating mantle is used in a traditional reflux apparatus. This is the equivalent of the stove a chef uses to provide the heat required to boil the soup. The shape and size of the heating mantles vary to fit the reaction vessel used in the refluxing. (Appendix A depicts a variety of commercially available scientific equipment used in the various manufacturing methods.)

All stoves have a method of regulating the heat they provide. Add too little heat, and the soup will not boil, or add too much heat, and the soup will boil over. A rheostat is used to regulate the heat produced by the heating mantle. It is plugged into any standard electrical outlet and is the equivalent of the control knobs on an electric stove. Regulating the amount of heat administered by the heating mantle controls the rate of the reaction.

Commercially available oil or water baths can be used as an alternative to a heating mantle. The reaction vessel is submersed partially or totally in the oil or water to provide a uniform source of heat around the reaction vessel when the reaction temperature is critical.

A chef needs a pot in which to cook his soup. A boiling or reaction flask acts as the pot. As a general rule, a boiling flask has a single opening, or neck. Reaction flasks have multiple necks. The multiple necks on a reaction flask have legitimate purposes during the refluxing process. However, all that is

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necessary for a basic reflux apparatus is a single opening. The opening allows the ingredients to be added, pressure to be vented during the reaction, and the product to be removed. Many clandestine recipes for controlled substances require a triple-neck reaction flask, while in reality, a single-neck flask would suffice.

Boiling and reaction flasks are generally spherical with round bottoms that fit snugly into the appropriately sized heating mantle. The flasks encountered in clandestine labs vary in size from 25 ml (slightly less than 1 oz) to 72 l (approximately 18 gal). There are also boiling and reaction flasks that have flat bottoms to allow the flask to sit on a hot plate or ring stand without support.

The necks on boiling and reaction flasks generally have ground glass fittings that provide a sealed connection for various auxiliary items, such as condensers, addition flasks, or thermometers. Smooth-necked flasks exist. Sealing connections with auxiliary equipment is accomplished by using cork or rubber stoppers.

A chef places a lid on his pot to keep the soup from boiling dry. The steam from the boiling soup condenses on the lid and drips back into the soup, replenishing the evaporating liquid. The steam escaping from around the sides of the lid relieves excess pressure. A reflux apparatus operates in much the same way. Various types of condensing columns or condensers act as the pot's lid. Each type has a specific scientific application. However, in the world of clandestine labs, all condensers act as the lid on the boiling soup.

A condenser has a compartment through which cool water is circulated. Steam from the boiling mixture condenses on the cool surface of the compartment. The condensed liquid then drips back into the boiling mixture. Vapors that are not condensed escape through an opening, relieving excess pressure on the system.

In some instances, the uncondensed vapors are vented into some sort of absorbent material, helping to eliminate the odors associated with the production method. This also traps the toxic vapors generated by the reaction. Unvented fumes containing toxic compounds can affect the health of the lab operators or personnel tasked with the seizure of clandestine labs.

Not using a condenser has many hazards. Uncondensed vapors fill confined spaces that house clandestine labs and create a toxic environment. A reflux apparatus without a condenser can potentially boil dry, creating a different set of toxic or hazardous compounds.

The venting process can be potentially hazardous. If the condenser becomes obstructed, pressure will build in the reaction vessel as a result of the boiling liquid. If the obstruction is not removed, one or more of several dangerous situations may occur. The pressure of the expanding gas could clear the obstruction violently. The expanding gas may compromise the con-

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nection between the condenser and the reaction flask, turning the condenser into a projectile. The structural integrity of the reaction flask or condenser may be compromised and result in a boiling liquid expanding vapor explosion (BLEVE).

Clandestine lab operators commonly create reflux apparatuses utilizing ordinary household items. Hot plates have been used as heating mantles. Countertop deep fryers have been used as oil baths. Glass cookware items have been used as reaction vessels. Condensers have been fabricated from copper or PVC pipes. The only limitation is the operator's imagination, so the clandestine lab investigator must also be thinking creatively in order to recognize things for what they really are.

1.3.1.2 Distillation

Distillation is the separation of a liquid from a solid or other liquid using evaporation followed by condensation. It is a modification of refluxing. It can be used as a technique to synthesize and separate compounds or used solely as a separation technique.

Distillation uses the differences in the boiling points of the mixture's components to separate them. The component with the lowest boiling point will separate from a boiling mixture first. The mixture will maintain the temperature of the boiling point of that component until it has completely evaporated from the mixture. The mixture's temperature will then rise to the next lowest boiling point in the mixture.

Knowing the boiling point of the component of interest allows the operator to isolate it. Even if the operator does not have a thermometer, he can isolate the desired component if he knows the boiling plateau of the component of interest.

During the distillation process, a mixture of chemicals is boiled. As a result, precursor chemicals can be converted into a desired end product or combined with other precursors to synthesize a new compound during the boiling process. At the same time, the unwanted by-products are separated from the mixture by evaporation.

The equipment used for distillation is the same as that used for refluxing. The individual components of the apparatus are rearranged to allow gravity to separate the condensing liquid from the boiling mixture rather than return it. As with a reflux apparatus, the distillation apparatus (Figure 1.6) requires a heating mantle (stove), a boiling and reaction flask (pot), and a lid (condenser). The heating mantle provides the heat required to boil the ingredients in the reaction vessel. The vapors from the boiling ingredients then condense in the condenser. The orientation of the condenser is changed to allow gravity to separate the condensing liquid away from the boiling liquid instead of returning it to the mixture. It may then be collected in a reception flask.

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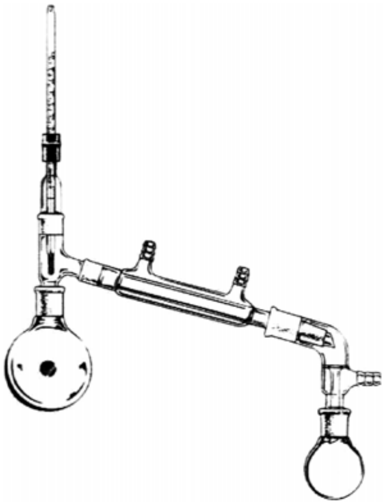

Figure 1.6 Simple distillation apparatus.

In simple distillation, the temperature of the boiling liquid is monitored to determine when the component of interest is evaporating from the mixture. Fractional distillation allows the separation of components with similar boiling points by monitoring the temperature of the vapors that traveled through a precooling (fractionating) column.

A vacuum pump may be attached to either system to aid in the distillation process. The vacuum pump draws the vapors into the condensing column rather than allowing the pressure from the boiling liquid to force them into the column. This expedites the process.

As with the reflux apparatus, clandestine lab operators often alter common equipment to create a distillation apparatus, when the need arises.

1.3.1.3 Hydrogenation

Hydrogenation is a chemical reaction that adds hydrogen to a substance through the direct use of gaseous hydrogen. Under high pressure, in the presence of a catalyst and hydrogen, ephedrine can be converted into methamphetamine. The hydrogenator (Figure 1.7) used in this method is commonly referred to as "the bomb" in scientific circles as well as in the clandestine lab world. Hydrogenators are commercially available. As with all scientific equipment, clandestine lab operators developed alternatives for this specialized piece of equipment.

1.3.1.4 Bucket Chemistry

There are certain manufacturing methods that do not require traditional chemical apparatuses. "Bucket" chemistry is an appropriate term, because these

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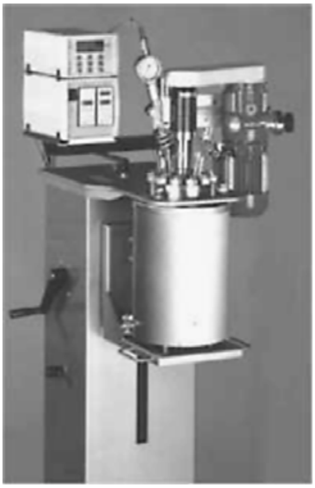

Figure 1.7 Commercial hydrogenator.

reactions can literally take place in a plastic bucket. The chemicals are placed into the container and allowed to react. At some point in time, an extraction process is undertaken to separate the final product from the reaction mixture. No heat is necessary, but cooling may be required. Phencyclidine and methamphetamine can be produced using nothing more than plastic containers.

1.3.1.5 Extractions

Extraction is the act of separating a constituent from the whole. It may be performed a number of times during the manufacturing process. Clandestine lab operators rely on the component's physical and chemical properties to isolate it from the rest of the substances.

The two types of extraction mechanisms are physical and chemical. The type does not necessarily correspond to the use of the component's physical or chemical properties, but it is simply a means of explaining the process.

Chemical extractions use a component's ability to dissolve in a liquid (solubility) to separate it from the bulk substance. The component being extracted may be the compound of interest or some unwanted by-product. The process does not require sophisticated equipment. All that is required is a container to hold the original mixture and a liquid that the desired material will not dissolve in.

Insolubility can be used as well. The salt forms of many drugs are not soluble in most organic liquids. When a free drug is converted into the salt form, it will precipitate out of an organic solution. During conversion of the drug from one form to another, its solubility properties are changed. This allows its extraction from the solvent and any by-products that are soluble in the solvent.

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The compound of interest does not have to be in a solid form to be extracted by a liquid. A liquid can extract other liquids from each other. The acidity of a liquid may or may not allow a substance to dissolve in it. For example, freebase methamphetamine is an oily liquid at room temperature. It is soluble in acidic water solutions but insoluble in basic solutions. Thus, by adjusting the acidity of the liquid, methamphetamine can be chemically extracted from a water solution.

Physical extractions physically separate the components of interest. In many instances, the act of chemically separating the desired component is only half the battle. In one manner or another, the two chemically incompatible components need to be physically separated. Specialized equipment was developed to perform this task.

On the other hand, decanting and evaporation are two means of physical extraction that do not require specialized equipment. Both techniques use differences in their physical states to physically separate components or mixtures.

Decanting is a simple form of physical extraction that separates liquid and solid mixtures. The solid material is allowed to settle to the bottom of the container. The liquid is then carefully poured from the container, disturbing as little of the sediment as possible.

Gravity filtration is a simple physical extraction used to separate solids from liquids. All that is required is a funnel, a filtering material, and a receptacle for the filtered liquid. The filter device is placed into the funnel, and the solid–liquid mixture is poured into the funnel. Gravity draws the liquid through the filtering material, leaving the solid trapped in the filter. This type of filtration can be slow and may not be conducive to removing solids of fine particle size.

The filter paper commercially made for gravity filtration applications is unnecessary. All that is required is something that will allow a liquid to flow through and keep the solid on the other side. In clandestine labs, coffee filters, sheets, and women's silk underwear have been found being used to filter solids from liquids.

Vacuum filtration is a method of expediting the filtration process (Figure 1.8). A vacuum is used to draw the liquid through the filtering material. It is an efficient method of separating liquid from solids and is most efficient for solids of fine particle size.

Commercial vacuum pumps are commonly found at the scene of clandestine drug labs. Clandestine lab operators may use alternatives, such as the compressor from a refrigerator or an air conditioner. Air compressors have also been replumbed to perform this function.

There are times during the manufacturing process when different types of liquids must be separated from each other. This is accomplished through

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Figure 1.8 Vacuum filtration.

the use of a separatory funnel (Figure 1.9). The liquid combination is placed into the separatory funnel, and the aqueous and organic liquids are allowed to form two distinct layers. The valve at the bottom of the funnel is opened, and the liquid is allowed to drain until the layer separation reaches the valve. The valve is then closed, and the desired liquid is saved for further processing.

There are alternatives to the use of the separatory funnel. The top liquid layer can be simply decanted off away from the top. Turkey basters have been used to separate one liquid layer from another. A water bottle with a squirt top has also been utilized as a makeshift separatory funnel.

Evaporation can be used to separate a solid that is dissolved in a liquid and does not require specialized equipment. The liquid is allowed to evaporate, leaving the solid. An outside source of heat can be used to accelerate the process.

Distillation is a form of evaporation used to extract one liquid from another. As previously discussed, it requires specialized equipment to evaporate, condense, and capture the different liquid fractions. The temperature should be monitored. However, the compound of interest can be isolated if its position in the sequence of boiling plateaus is known.

There are some instances in which chemical and physical extractions are used in tandem. In the final purification stage of production, the salt form of a drug is placed into a funnel containing a filtering material. The solvent

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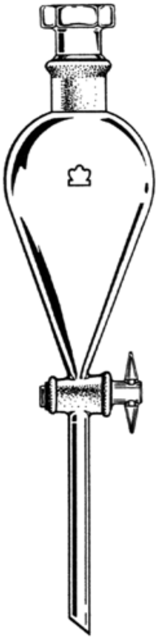

Figure 1.9 Separatory funnel.

is removed from the final product through a filtration process. Then, a different solvent, one in which the final product is not soluble, is poured onto the substance. This liquid chemically extracts the by-products from the final product and is physically extracted from the solid by filtration.

1.3.2 Chemical Needs

The chemical needs of a clandestine lab make up the second leg of the triangle. Most of the chemicals used in the manufacture of drugs and explosives have legitimate uses. Many can be obtained without restrictions through chemical suppliers or from grocery, drug-, or hardware stores. Some of the chemicals with restrictions have legitimate alternatives or sources that do not have restrictions on their distribution.

Some clandestine lab operators have a pragmatic solution: if they cannot legally buy a chemical, they will make it. A case demonstrating this philosophy occurred in Arizona, where a clandestine lab operator was utilizing phenylacetic acid as a starting material in a methamphetamine synthesis. Phenylacetic acid is a regulated chemical. To circumvent this problem, the operator manufactured it using mandelic acid as a starting material. He converted the mandelic acid into phenylacetic acid and continued the synthesis from there.

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All of the chemicals used in the manufacture of controlled substances have legitimate industrial uses. Some have legitimate home and hobby uses and may be found in anyone's kitchen, medicine cabinet, garage, or workshop. The key to forensic clandestine lab investigations is the ability to recognize combinations of chemicals that can potentially form a controlled substance.

Appendix B lists chemicals utilized in the manufacturing of controlled substances and their legitimate uses. Also noted are legitimate home or hobby uses for each chemical. This information can be used by the investigator to establish whether a reasonable person would deduce that there is a legitimate reason for the chemical to be at a particular location or in the possession of a given person. A variety of clandestine uses for a variety of chemicals in the manufacture of controlled substances are listed in Appendixes C and D. In Table A of Appendix C, generic indications of what certain chemicals can potentially be used to manufacture are presented. Related in Table B of Appendix C are specific combinations of chemicals used to manufacture controlled substances.

When obtaining a search warrant, investigators should be able to articulate that the chemicals known to be associated with the clandestine lab have legitimate uses but have no legitimate home or hobby use. Further, an investigator should be able to state which controlled substance this particular combination of chemicals may produce.

The three types of chemicals used in the manufacture of controlled substances are precursors, reagents, and solvents. All three types are used at some point during the manufacturing process.

A precursor chemical is a raw material that becomes part of the finished product. It is the building block with which the final product is constructed. In a conversion reaction, a precursor's chemical skeleton is altered to create the final product. In the synthesis process, precursors are chemically bonded together to produce the final product.

Reagent chemicals react chemically with one or more of the precursor chemicals but do not become part of the final product. During the process, a portion of the reagent may be part of an intermediate product but is removed prior to the formation of the final product.

The use of magnesium in the manufacture of PCP is an example. The magnesium reacts with bromobenzene to form the intermediate product phenymagnesium bromide. This intermediate product reacts with another intermediate product, phenycyclohexyl carbonitrile (PCC), to form phencyclidine (PCP). During the process, the magnesium is removed from the intermediate and returned to the reaction mixture solution.

A solvent does not chemically react with precursor or reagent chemicals. Solvents are used to dissolve solid precursors or reagents, to dilute reaction mixtures, and to separate or purify other chemicals.

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Some of the chemicals perform dual roles. For example, hydriodic acid (HI) acts as a reagent and as a solvent in the reduction of ephedrine to methamphetamine. This is because HI is not pure hydrogen iodide. It is technically a solution of hydrogen iodide and water. The hydrogen iodide acts as the reagent chemical. The water acts as the solvent in which the reaction takes place.

In the ephedrine/HI reaction, the water in the hydriodic acid (HI) acts as the solvent. It does not become part of the final or intermediate products. It only provides the environment necessary to allow the iodide from the hydrogen iodide to attach to, and be removed from, the ephedrine molecule to form methamphetamine.

An organic liquid can act as a solvent during extractions. The desired compound dissolves in the liquid and can be separated from the bulk substance. No portion of the solvent becomes part of the compounds being extracted.

Many chemicals have a variety of names. Many have a common name along with an official name chosen by the International Union of Pure and Applied Chemists (IUPAC). Common clandestine lab chemicals also have one or more slang terms (that may vary regionally) associated with them. For example, 2-propanone is commonly referred to as acetone. Therefore, investigators should rely on a forensic chemist to sort the various chemical synonyms.

Pronunciation of chemical names and terms can also be a problem. An investigator verbally asked a chemist what “propozyfene” was. The chemist had never heard of the compound. It was not until the investigator spelled the compound that the chemist realized that the investigator was trying to pronounce Propoxyphene, a Schedule II narcotic drug. For this reason, it is suggested that when nontechnical personnel attempt to describe or identify chemicals, they either write the name of the chemical or select the chemical name from a prepared list of chemical names and synonyms.

1.3.3 Knowledge Needs

Knowledge is the final leg of the manufacturing triangle. Knowing how to combine the equipment and chemicals to produce a controlled substance is a necessary element. Knowledge is necessary to establish capability and criminal intent. Knowledge may come from education (schooling or professional training), mentoring/apprenticeship, underground literature, and often even simply handwritten recipes that are bought and sold as property. The Internet has unfortunately become a source for many “recipes” as well. Original methods were taken from academic chemical literature and translated into simple recipes that can be followed by someone with no chemical training.

There are numerous ways to manufacture any given controlled substance. Each method has its roots in legitimate chemical or pharmaceutical litera-

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ture. An example of this was found in a clandestine lab that converted mandelic acid to phenylacetic acid that was then used as a precursor to methamphetamine. The origin of the recipe was tracked to a German pharmaceutical journal. The chemical proportions utilized by the recipe differed by a factor of 10 from the original article. The steps utilized in the reaction sequence were a translation from technical jargon into simple English (e.g., "reflux" in the original article was "boil" in the recipe, and "decant" was changed to "pour").

Underground literature is a large source of the knowledge used by clandestine lab operators. Books like *The Secrets of Methamphetamine Manufacture* (for drugs) and *The Anarchist's Cookbook* (for explosives and booby traps) have long been staple sources of "how-to" information. The information in these books originated in legitimate chemical research, and in some cases, the underground authors even cite the original source of the information. The scary part of this plagiarism is that these are often poor translations; lives are put in danger at every step of the process.

In the past, when the Leukart reaction was the methamphetamine manufacturing method of choice, clandestine lab operators served apprenticeships under experienced "cooks" to learn the tricks of the trade. Recipes were guarded zealously and bought and sold as commodities. Today, with the free flow of information over the Internet, these recipes can be obtained by anyone with a computer and a modem. There are Web sites dedicated to drug and explosive manufacture. There are newsgroups in which an operator can shop for a new recipe or source of chemicals and equipment: a strange sort of support group for the underground chemist.

The downside to this information source for the underground chemist is that all of the information is not good. Some of the recipes do not produce the desired product. Other recipes may explode when the operator follows the instructions provided.

One example of a recipe that does not work is the chickenfeed recipe for methamphetamine. There has long been a rumor that several manufacturers of chickenfeed place methamphetamine in their product to enhance the egg-laying capability of the chickens. If the clandestine lab operator follows a simple extraction process outlined in the recipe, so it goes, he should be able to isolate the methamphetamine. The only problem with the recipe is that the chickenfeed manufacturers do not place methamphetamine in their product.

Some states have a statute that makes the possession of chemicals and equipment for the purpose of manufacturing a controlled substance illegal. This can be problematic in that there are methods of manufacturing methamphetamine that utilize equipment and chemicals that can be commonly and legitimately found in any home in the United States. If this statute were

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interpreted literally, thousands of homes in the United States would be in violation of the statute. Just because there is over-the-counter cold or diet tablets, rubbing alcohol, iodine solution, swimming pool acid, glass jars, a turkey baster, and coffee filters at a location does not make it the site of a clandestine lab. In order to prove intent, the knowledge must be proven to be present as well. Did the person who possessed the items know how to combine them in the proper sequence to make a controlled substance?

Knowledge, alone, is not all that is required. If that were the case, half of the chemistry students at any given university would be in jail. The key to the statute is the words "for the purpose of." Did the person with the equipment, chemicals, and knowledge intend to combine them to create a controlled substance? This is the question that needs to be asked when evaluating the knowledge requirement. In essence, did the person have the requisite criminal intent?

1.4 Summary

Clandestine labs come in all shapes and sizes. Lab operators range from the small-scale operator who produces just enough to sustain his personal habit to the large-scale operator who produces pounds at a time for commercial profit. Clandestine labs are found in every segment of society and cross all demographics. Race, religion, age, sex, and economic status are neither indicators nor barriers.

All three legs of the clandestine lab triangle need to be present for a clandestine lab operation to exist. Equipment, chemicals, and knowledge must be present for the operator to produce the desired product. The equipment used ranges from technical and scientific to mere household kitchen utensils. The chemicals required vary from the exotic that are only available through scientific supply houses to those that can be purchased over the counter at any drug-, grocery, or hardware store. Even though a science degree is helpful, all a clandestine lab operator needs to be able to do is follow directions.

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Clandestine Lab Hazards

2

Clandestine lab investigation is one of the most dangerous tasks undertaken by law enforcement. The dangers go beyond the violence of the suspects involved in the illegal operation. There are seen and unseen hazards with these toxic operations that must always be kept in the forefront of the minds of the personnel involved in investigation and seizure. The effects of some of these hazards on a human being may not be seen until long after they were encountered.

In this chapter, the different hazards associated with the seizure of clandestine labs are presented. A general overview of the different types of clandestine lab hazards will be presented. While some sections may be a simplified refresher for experienced investigators, the goal is for everyone involved in clandestine lab investigations and prosecutions to understand the scope of the hazards involved.

2.1 General Hazards

The three things clandestine labs have in common, regardless of their location or sophistication, are the simple facts that the operators usually have little chemical training, the operations are makeshift, and no two operations are alike. These three principle hazards exist, whether the final product is methamphetamine or PCP, flash powder or nitroglycerine.

2.1.1 Little Training

With the exception of the educated operator mentioned in Chapter 1, clandestine lab operators have little chemical background or training. Their for-

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mal education is limited either in years or content. The so-called "Mexican National" labs, operating in the western United States, are a good example. Illegal aliens are hired to tend methamphetamine production operations. They have little education and do only as they are told. They do not understand the physical and chemical principles of the reaction that they are tending, much less the hazards involved.

The lack of chemical training can lead to hazards within the clandestine lab that endanger more than just the operators and the enforcement personnel tasked with seizing the operation. Unsuspecting people in the area adjacent to the clandestine operation are exposed to many of the same hazards, often with disastrous consequences. There are numerous examples of lab operators, enforcement personnel, and innocent bystanders being hurt as a result of actions taken by clandestine lab operators who do not understand the principles of chemistry and physics that govern the chemical reactions that occur within the operation. Houses and apartments have been destroyed by fire. Explosions were caused by operator error. Emergency responders and neighbors were exposed to toxic fumes generated by the reaction of incompatible chemicals.

Clandestine lab operators neither understand nor practice common laboratory safety procedures (Figure 2.1). They are notorious for storing incompatible chemicals together; strong acids and strong bases are commonly found stored adjacent to each other. Organic acids are stored with oxidizing acids. Flammable liquids are stored near a source of ignition. Chemicals are routinely unlabeled or mislabeled to avoid detection. Waste material from reaction mixtures is often combined without regard to content or pH.

The improper storage and handling of chemicals may lead to violent chemical reactions between incompatible chemicals, or the chemicals may react and create substances that are more toxic than the original chemicals. Emergency medical service personnel often encounter operators at clandestine lab sites who exposed themselves to the toxic waste or by-products of the combination of incompatible chemicals in reaction mixtures or waste material. Improper handling of the chemicals leads to human exposure of unknown chemical hazards, which makes it difficult to treat the exposed operator.

An example of mixing chemicals to create a toxic atmosphere occurred when an operator mixed a waste solution containing acid with a solution containing sodium cyanide. The resulting hydrogen cyanide placed the operator, one police officer, and two EMS responders in the hospital. Fortunately, no deaths occurred in this scenario.

The concepts of vapor pressure, flash point, flammability, and explosive limits are not usually in the operator's knowledge base. Nationally, approximately 20% of clandestine labs are detected as a result of fire or explosion. Flammable vapors build up inside the lab space, reaching the flammable or

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(a)

(b)

Figure 2.1 (a) Ether in trunk. (b) Abandoned explosive chemicals.

explosive limit of the chemical involved. The operator lights a match, turns on a gas stove, or turns on a light switch, thus igniting the fumes in the lab and resulting in a fire or explosion.

Figure 2.2 shows the results of an attempt to evaporate methanol from an extraction solution containing pseudoephedrine. The operator used methanol to extract the pseudoephedrine from an over-the-counter cold preparation. He placed the methanol solution on a gas stove to speed the evaporation process. The combination of the methanol vapors and the gas flame resulted in a fire that caused extensive damage to the two-bedroom bungalow. The operator was caught a week later, when he again caused a vapor explosion under similar circumstances in a motel room less than 2 miles from the location of the bungalow.

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(a)

(b)

Figure 2.2 Fire from methanol evaporating on a gas stove. (a) Gas stove fire point of origin. (b) Burned methanol container.

2.1.2 Makeshift Operations

The creativity of clandestine lab operators is amazing. Using a basic understanding of how scientific equipment operates, they design equipment alternatives that allow them to avoid detection by not using scientific supply houses as equipment sources. Also, the cost of the homemade alternatives can be significantly less than the actual items.

The problem with this situation is that even if the operator has taken the physics and mechanics of the equipment into account, he probably has not taken into consideration the interaction between the chemicals involved and the materials with which the makeshift equipment is constructed. Common glass kitchen utensils cannot be substituted for Pyrex™ glassware that is designed to operate at high temperatures. Rubber or cork stoppers cannot be substituted for ground glass connections in reflux and distillation appa-

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ratuses when using organic solvents or strong acids. Reaction vessels made of steel are not designed to contain solutions of hydrochloric acid.

In Figure 2.3, three examples of the ingenuity of clandestine lab operators are shown. Shown in Figure 2.3(a) is a countertop deep fryer being utilized as an oil bath heat source for a reflux operation. Shown in Figure 2.3(b) is a hydrogenator that was constructed out of a beer keg and heat tape. In Figure 2.3(c), a homemade condenser made from different diameters of copper tubing is shown. Yet, each of these alternatives efficiently accomplished the task for which it was designed.

2.1.3 No Two Labs Are Alike

There are hundreds of different methods of manufacturing controlled substances. However, only a small number of methods are actually encountered. Even when the same method is repeatedly encountered, there are still enough differences within each clandestine lab to make each unique.

It cannot be repeated emphatically enough that the personnel responding to the scene of a clandestine lab must be constantly vigilant for potential hazards. Even though the particular type of operation may have been encountered numerous times before, it should always be treated as an unknown scenario. Complacency is the biggest danger to personnel investigating or processing the scene. A less-than-aware attitude may lead to not recognizing booby traps, disregarding the presence of odd or unique chemicals, exposing oneself to toxic environments, or improperly handling unsafe makeshift equipment setups. It can further lead to severe injury for the investigators.

2.2 Hazard Priority

There are numerous hazards associated with clandestine labs. These hazards were grouped into priority categories according to the immediate harm they can present the personnel responding to the scene of a suspected clandestine lab. The hazard groups are, in order of priority, explosion, fire, firearms, and exposure.

2.2.1 Explosion

An explosion is a rapid chemical change that produces a large amount of heat and gas. It is the highest hazard priority, because it can potentially do the greatest amount of damage to the responding personnel in the shortest amount of time. The source can be intentionally placed (i.e., a booby trap) or an unintentional result of the improper handling of chemicals.

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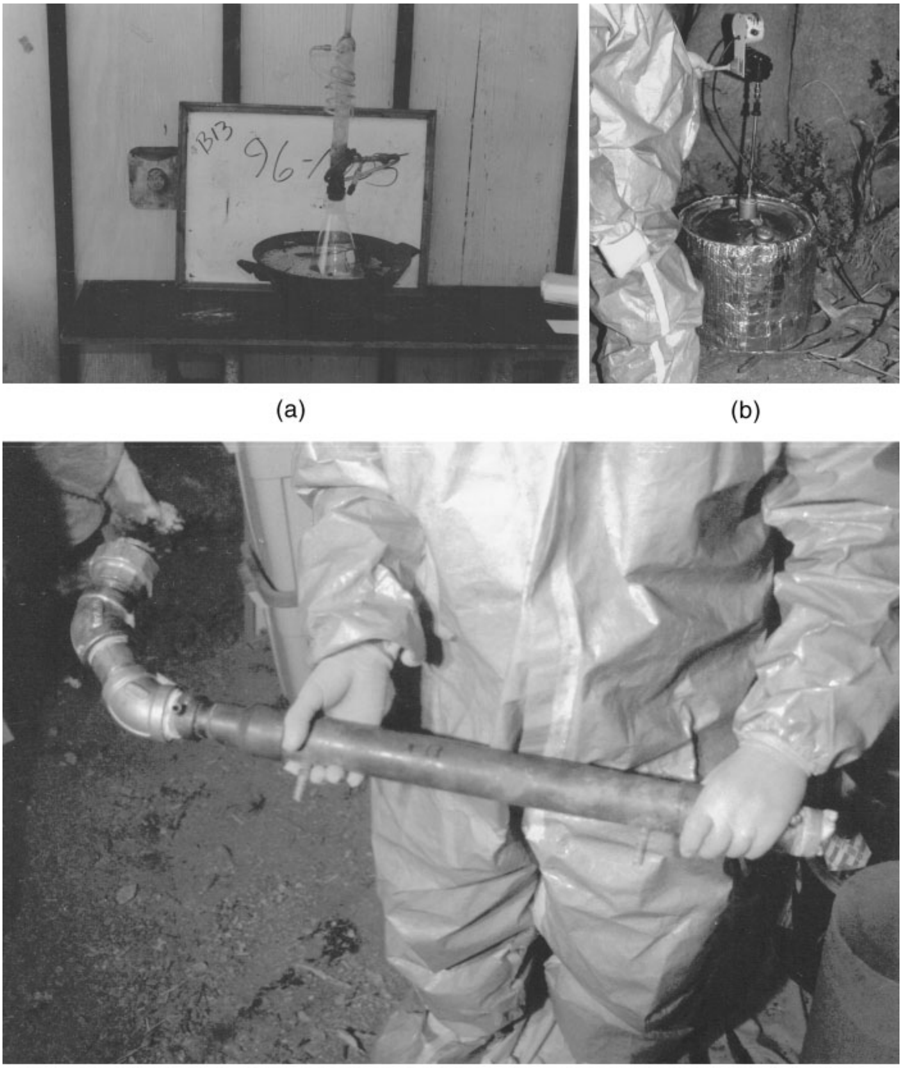

(a) (b) (c)

Figure 2.3 (a) Oil bath reflux. (b) Beer keg reaction vessel. (c) Copper tube condenser.

Explosions have two effects on the body. First, the resulting pressure wave can cause internal and external damage to the body by the blast directly or by the secondary effects resulting from being struck by items thrown as a result of the explosion. The second effect is a result of the high temperatures created by the explosion. Both of these effects are reduced for the exposed person as the distance away from the center of the explosion increases.

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The three types of explosions are detonations, deflagrations, and mechanical explosions. The only significant difference between the first two is the rate of the reaction. All have pressure waves and intense heat associated with them. The pressure wave of the explosion is proportional to the amount of energy released, which determines the effect on the body that is exposed to the explosion.

Detonations are chemical reactions that produce extraordinary amounts of heat and gas, resulting in a pressure wave that causes the damage. They are the result of known explosive materials or chemical mixtures that have a reaction rate of greater than 1000 miles per second. They can also be the result of the reaction between a mixture of incompatible chemicals or the undesirable outcome of the mishandling of unstable chemicals, such as peroxides, white phosphorus, or picric acid.

Deflagrations have a reaction rate of less than 1000 miles per second. They are explosions that result from the pressure created by a chemical reaction, usually combustion, that compromises the structural integrity of the container. They can result from the spontaneous ignition of an atmosphere that contains ignitable liquid vapors in an explosive concentration, if the ignition takes place in a contained environment (i.e., a room). Confined deflagrations of combustible solids, such as smokeless powder, can also produce explosive results.

Mechanical explosions result from a pressure buildup in a container to the point at which it loses its structural integrity. A boiling liquid expanding vapor explosion, or BLEVE, is an example. The expanding vapors of a boiling liquid in a closed container create so much pressure that the container explodes. In practical examples presented in Chapter 9, the effects of explosion in an actual setting are demonstrated.

The lack of chemical knowledge of the clandestine lab operator can create a situation that is prone to explosive results. Ethers are exposed to the atmosphere, creating unstable peroxide compounds. Picric acid stored in containers with metal lids and allowed to dry will form metal picrates on the treads of the lid that can explode when the lid is twisted. Metallic sodium or lithium stored improperly can explode when exposed to air or water. Flammable vapors may be allowed to collect in a confined space, and when ignited, they will deflagrate, producing explosive results.

Figure 2.4 shows examples of improperly stored chemicals that pose an explosive hazard. The jar without a label, shown in Figure 2.4, contains dry picric acid. The operator placed the label inside the jar so he would know what the contents were.

Other causes for explosions are intentional on the part of the operator. According to DEA statistics, approximately 10% of clandestine labs are booby-trapped. These booby traps may or may not be directed at law enforcement.

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Figure 2.4 Explosive chemicals.

No matter whom the target, personnel processing clandestine lab scenes must be constantly aware of the potential existence of explosive devices.

The abatement of explosive hazards is accomplished by explosive ordinance disposal (EOD) experts and chemists trained in the clandestine manufacture of controlled substances. They should jointly process all clandestine lab sites for the presence of explosive devices and dangerous chemical mixtures prior to processing the site for physical evidence. The EOD expert and chemist should have complimentary knowledge of each other's expertise. This team should be able to observe, recognize, and neutralize potentially explosive situations before the rest of the processing team enters the location.

Shown in Figure 2.5 is an example of a vapor explosion that occurred in a gamma hydroxybutaric acid (GHB) lab. Shown in Figure 2.5(a) is a demonstration of the pressure effect of the explosion. The wall at the center of the explosion was blown approximately 3 ft off center. The resulting fire damage is shown in Figure 2.5(b). In this scenario, the operator was drying acetone from his finished product in a heated room (confined space). The acetone fumes reached their explosive limit. When the operator turned on the light in the room, the spark that occurred when the light switch was thrown ignited the vapors, causing the explosion.

2.2.2 Fire

Fire is second in the priority list of hazardous situations the clandestine lab investigator must consider. It has many of the same causes as explosive hazards. Booby traps, ignition of flammable atmospheres, incompatible chemical mixtures, and the operator can contribute to the cause of a fire. A significant difference between fire and explosive hazards is lack of the pressure wave experienced from explosions. However, the heat and resulting combustion damage can be similar.

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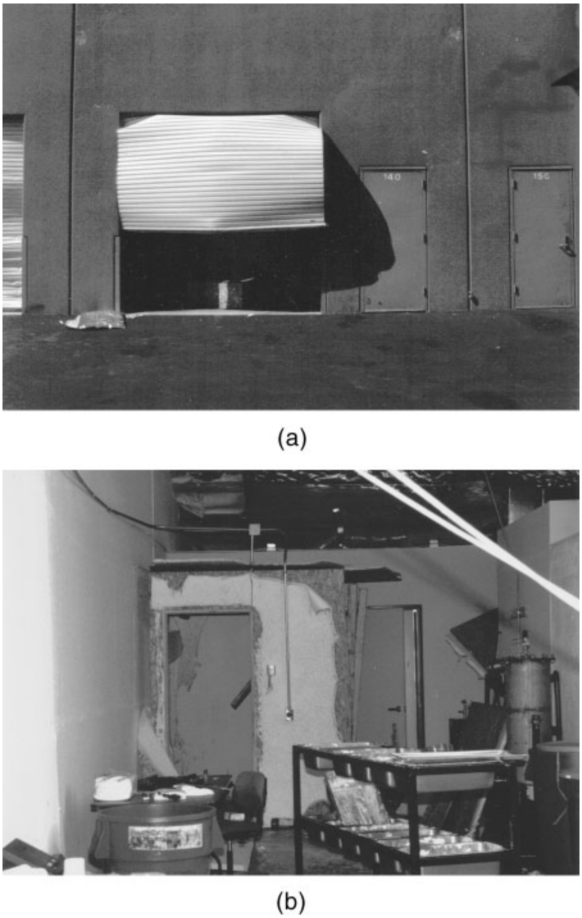

Figure 2.5 Images of a GHB lab. (a) Vapor explosion effect on entrance door. (b) Fire damage on the drying room.

Flash fires and sustained fires are the two types of fire hazards. Flash fires result from the ignition of a flammable atmosphere. They are instantaneous, and usually self-extinguish for lack of fuel. If contained, a flash fire may result in an explosion or produce minor pressure-related effects. A sustained fire, on the other hand, has a continuous source of fuel to feed the fire. The resulting heat and continuous flame allow the fire to spread beyond the area of the original ignition.

Test results from the Phoenix, AZ, Fire Department indicated that exposure to a flash fire may be survivable. If the victim were wearing fire-resistant clothing, significant damage would be limited to the unprotected areas. If the victim was not wearing fire-resistant clothing, the severity of the burns to the protected areas of skin would be determined by the type of clothing

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he was wearing. The test dummy wearing fire-resistant clothing did not fare as well when exposed to a sustained fire situation, during the same series of tests. The clothing was consumed by fire when exposed to a sustained fire of flammable liquids commonly encountered in clandestine labs.

As with explosions, clandestine labs are commonly detected as a result of a fire. Figure 2.2 showed an example of the results of a fire caused when the lab operator attempted to evaporate a flammable liquid (methanol, in this case) on a gas stove. The fire could have been started just as easily if the flammable liquid had been on an electric stove. Any ignition source would have ignited the flammable vapors. Once the concentration in the room reaches the flammable or explosive limit, a spark from a light switch, a match struck for a cigarette, or a muzzle flash from a discharging weapon could ignite the vapors.

The images in Figure 2.6 show the result of a barricaded clandestine lab operator intentionally burning his lab, in an effort to avoid detection. The operator could just as easily have waited until the investigators were inside the location before he ignited the flammable and combustible substances within the operation, causing incredible injury.

2.2.3 Firearms

As described, the clandestine lab investigator has more things to worry about than just getting shot, but that still ranks up there with dangerous situations potentially encountered.

Individuals who abuse methamphetamine develop a paranoid psychosis. These individuals think their personal safety is continuously under attack. As a result, they can be unpredictable, violent, and, in general, are thus likely to be armed with some type of weapon (Figure 2.7). These people are irrational and subject to delusions and hallucinations.

The threat of firearms is not over once the suspects have been arrested and removed from the scene. It is common for armed secondary suspects to appear at the scene once the processing begins. This is a real hazard because many of the people processing the scene are not authorized to carry weapons. Even those who are may not have them in their possession.

2.2.4 Exposure

Exposure to chemical and physical hazards is lowest in the priority of hazards. This does not mean that it is any less dangerous to the clandestine lab investigator. These are silent hazards. Their effects may not become apparent until after the exposure.

Exposures to chemical and physical hazards may have acute and chronic effects. Acute effects are experienced by exposure to hazards of high concen-

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(a)

(b)

(c)

Figure 2.6 Images (a), (b), and (c) show the progression of an intentionally set fire.

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Figure 2.7 Guns on the wall of a clandestine lab.

trations, even for a short duration. The effects are felt immediately. The exposed generally will recover if the exposure does not exceed lethal limits. Chronic effects are generally experienced by numerous exposures to chemical hazards in low concentrations over long periods of time. The effects are cumulative, creating a toxic effect. These effects are usually different than the acute effects of the same hazard.

The difference between acute and chronic effects can be demonstrated in the example of a person who gets drunk and falls every night for an extended period of time. The acute effects of the alcohol may cause the person to lose control of his motor functions, causing him to fall and hit his head. Hitting his head may have one acute effect — that of a fierce headache — but the effect is short-lived. The person eventually sobers up, and the headache goes away.

A chronic effect of drinking alcohol is different. Long-term exposure to alcohol may lead to cirrhosis of the liver; brain damage can result from

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repeated blows to the head over an extended period of time. These effects will not go away by simply removing the alcohol from the person after the fact.

2.2.4.1 Chemical Hazards

All chemicals are hazardous. The degree of hazard depends upon the chemical's properties, the proximity to other chemicals, and the other chemicals' properties. While water is considered a benign substance, when exposed to sodium metal, the combination becomes explosive.

Many common household products contain hazardous chemicals that can be used to manufacture controlled substances and explosives. Acids can be found in swimming pool chemicals and battery acid. Bases or caustic compounds are commonly used in drain cleaners. Flammable solvents found in paint thinners and camping fuels are used for extractions. Poisonous chemicals used as automobile antifreeze are the precursor for an explosive. Just because these chemicals are found in the home does not make them any less hazardous.

The hazardous properties a chemical can possess are varied: explosive, flammable/combustible, corrosive, oxidizing, compressed gases, and poisonous. The chemical's reactivity potential should also be taken into account. A single chemical may possess multiple hazardous properties. For example, nitric acid (HNO_3) is a corrosive, oxidizing poison. Listed in Appendix E are the hazards associated with chemicals commonly encountered in clandestine labs.

Explosive chemicals undergo rapid chemical changes that release large amounts of heat and gas. This rapid chemical change may result from encountering a shock or friction, being exposed to a source of ignition, or being subjected to sudden changes in temperature, water, or air. Certain chemical combinations will explode, or a mixture will be created that will explode at the least provocation.

It is rare to encounter explosive chemicals, unless the operation's final product is an explosive. However, there are numerous combinations of chemicals that will explode under the proper conditions. That is why it is imperative that properly trained EOD personnel and chemists evaluate clandestine lab sites prior to evidence collection.

Most organic chemicals will sustain combustion if exposed to an ignition source or to enough heat. The chemicals with the greatest fire hazard are those that ignite easily at low temperatures. Flammable liquids are those that have a flash point below 100°F. Combustible liquids are defined as those having a flash point above 100°F. Combustible chemicals, such as phosphorus and magnesium, are solids that can easily sustain combustion (Figure 2.8).

Many of the solvents used for extraction or purification purposes are extremely flammable. Solvents like methanol and acetone have low flash

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Figure 2.8 Combustible solid mixture.

points and readily ignite; these chemicals also have the potential to create a flammable or explosive atmosphere. Solvents like xylene or mineral spirits burn, but because of their higher flash points, are less likely to form a flammable or explosive atmosphere.

Corrosive chemicals are those that can cause visible damage to metals, plastics, or other materials (especially your skin). They are composed of acids and caustic or basic compounds. Acids are compounds that readily donate a proton (hydrogen) to a chemical reaction. They have a pH less than seven (with a pH of two or less considered corrosive). They can be subdivided into organic and mineral acids. Organic acids are compounds that contain carbon. Mineral acids do not contain carbon but may (oxidizing) or may not (non-oxidizing) contain oxygen.

Acids are soluble in water (Table 2.1). In concentrated solutions, they will attack minerals and tissues. They can coagulate protein. Contact with oxidizing acids or a reaction with organic material can result in fire. Metals reacting with sulfuric, nitric, or hydrochloric acids can create an explosive environment.

Acids are generally found in synthesis and conversion labs. They are used as reagent chemicals. The acid alters the chemistry of the compounds and provides the physical environment necessary for the reaction to take place. Acids are also used to convert freebase drugs into the water-soluble salt form that is sold to the end user.

Acids generally exist in a water solution of varying concentrations. Pure acids are rarely encountered. However, mineral acids, such as hydrochloric acid and hydriodic acid, produce fumes. These fumes can fill the atmosphere

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Table 2.1 Acid Relative Strength

Acid Name	Formula	
Perchloric acid	$HClO_4$	Stronges t
Sulfuric acid	H_2SO_4	
Hydrochloric acid	HCl	
Nitric acid	HNO_3	
Phosphoric acid	H_3PO_4	
Hydrofluoric	HF	
Acetic acid	CH_3COOH	Weakest

in and around the lab area. As a rule of thumb, "hydro-" acids are corrosive and will produce fumes.

For identification purposes, if the label says acid in the chemical name, it should be considered corrosive. The strength and concentration may be unknown, but the compound will react like an acid. Therefore, treat all unknown acids as if they were extremely corrosive.

Caustics or bases are chemicals that readily accept a proton (hydrogen) in a chemical reaction. They have a pH greater than seven with any pH greater than 12 considered corrosive. They can be subdivided into inorganic peroxides and organic amines. Inorganic peroxides are characterized by the presence of a hydroxyl ion (OH^-) group. The organic amines contain a characteristic amine ($-NH_2$) group.

Caustics generate heat when reacting with water, acids, organic material, and some metals. They have a tendency to liquefy protein (i.e., tissue). Caustics are used to neutralize acids in the synthesis and conversion processes. They are used to adjust the pH of water solutions in the preparation of an extraction. In conversion labs, they are used to convert the salt form of a drug into the freebase form. In some instances, they can act as a precursor chemical and become part of the final product.

Caustics can be found in solid, liquid, and gas forms. They are found in pure forms more often than their acidic counterparts. However, they can be just as easily found in a water solution of varying concentrations. Words like caustic, hydroxide, and amine in the chemical name indicate a compound with caustic or basic properties.

Oxidizers are compounds that provide oxygen to a reaction. These compounds may cause a fire if they come in contact with combustible material, and they can react violently when exposed to water or in a fire. Oxidizers contribute oxygen to chemical reactions, which increases the fire and explosion hazard, because they provide a source of oxygen to sustain combustion in a normally oxygen-deficient atmosphere. An excess amount of available

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oxygen can also increase the reaction rate, making the combustion hotter and faster.

Oxidizers are used as reagent chemicals in the manufacture of drugs and explosives. More significantly, they are a major component of inorganic explosive mixtures. Oxidizers are generally found in solid forms. Some strong acids, such as nitric acid and sulfuric acid, act as oxidizers. Compounds with names ending in "-ate" (i.e., chlorate, nitrate, permanganate) are compounds with strong oxidizing potential.

As acids have bases as a conjugate, oxidizers have reducers. A reducer is a compound that can remove oxygen from or add hydrogen to a compound. Reducers are used as reagent chemicals in conversion labs. Strong reducing agents react rapidly and violently. They are found in solid and liquid forms, and their labels contain the word hydride or acetylide in the chemical name.

Compressed gases pose a dual hazard. Not only does the chemical inside the container have unique chemical hazards associated with it, but also, the container can pose a threat. The incompatibility of the contents with its container may cause the container to explode or discharge its contents unexpectedly. This often happens in situations in which the operator refilled a container with something other than what the container was designed for.

There is no accepted color code for compressed gas cylinders. The color of a compressed gas cylinder containing helium may vary depending upon the distributor. The only clue as to the contents of a legitimate gas cylinder is the connection on the top. For example, compressed gas cylinders to be used for compressed air have unique fittings, so a flammable gas cannot be accidentally connected to the line.

In the world of clandestine labs, it is a given that the outside label of a container may not reflect the contents. Operators routinely replace the contents of compressed gas containers with chemicals other than those the container was designed to hold. These replacement chemicals will often corrode the brass fittings used to regulate the release of the compressed gases. This creates a hazard for anyone handling the container. Even the minor act of opening the valve may cause it to break, releasing the pressurized contents in a single violent rush. When the contents are unknown and may be under high pressure, and there is generally no safe way to sample the contents in the field, the field investigator may consider not to even sample the contents of the compressed gas cylinders and to simply allow the chemical waste disposal company to dispose of them properly.

Another compressed gas hazard is the homemade hydrogenator (Figure 2.9). This apparatus is made of materials that were not designed to withstand the pressures or temperatures generated by the reaction. The minor act of touching the container may be enough to compromise the structural integrity

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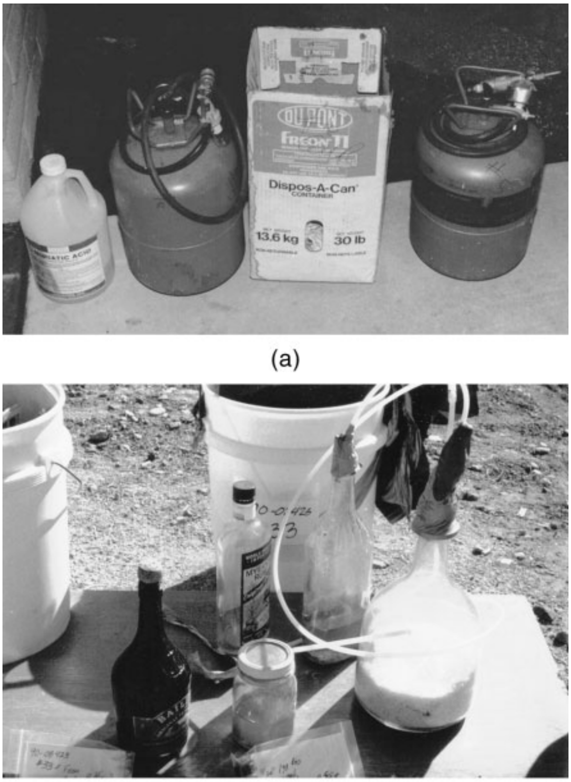
of the container if it is under pressure, causing a BLEVE-like explosion that sprays the contents of the container over the surrounding area.



(a)

(b)

Figure 2.9 (a) Compressed gas container. (b) Clandestine HCl generators.

A poison is a substance that in low concentrations will cause death or injury upon ingestion. Poisons act on the internal systems of the body. Highly toxic poisons are usually gases or highly volatile liquids that have an oral LD50 of <5 mg/kg. Moderately toxic poisons may be solids or liquids, with an LD50 of >5 mg/kg.

Chemical reactivity is the final hazard. How chemicals interact with each other can potentially pose a significant threat to clandestine lab responders. How the responders handle the chemicals at the scene is just as important as what lab operators did with them prior to their arrival.

Chemicals are either compatible or incompatible. Compatible chemicals can remain in close or permanent contact without a reaction. Incompatible chemicals react with undesirable results.

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Incompatible chemical reactions generate heat that can cause a fire or explosion, form a toxic gas or vapor, or form a substance that is more toxic than the original compounds. The reaction can disperse a toxic mist or dust, produce a violent chemical reaction, or any combination thereof.

Water-reactive and pyrophoric chemicals are examples of chemical groups that pose extreme reactivity problems. Water-reactive chemicals hydrolyze with water, forming flammable, corrosive, or toxic products. Pyrophoric chemicals react with the air and may spontaneously ignite.

Metallic sodium used in the Birch reduction is an example of a water-reactive chemical. It reacts violently with water, producing sodium hydroxide and hydrogen gas. Sodium hydroxide is extremely caustic. Hydrogen is extremely flammable and, under the right conditions, explosive. Under the right conditions, the water reacting with metallic sodium can produce an explosive hydrogen environment. If the hydrogen explodes, a caustic aerosol of sodium hydroxide would be dispersed. For this reason, metallic sodium is stored in mineral spirits of other nonaqueous solvent.

2.2.4.2 Physical Hazards

The final group of hazards associated with a clandestine lab is the physical hazards. These hazards include accidents, thermal exposure, electrical dangers, and the dangers of confined spaces.

An accident is an unforeseen happening resulting in damage to people or property. Accidents are not the result of an unsafe act. For example, if a person opens a door without realizing there is someone on the other side, and the door hits the other person, causing them to spill red wine on their shirt, the person opening the door did not know there was anyone on the other side and could not foresee the collision; therefore, it was an accident.

On the other hand, if an investigator opens an unknown chemical container without the appropriate training or personal protective equipment, spilling acid on his shirt, damaging it and burning his skin, this was not an accident. The actions and damages were avoidable. The investigator should not have been handling the container without the proper training, or minimally, without the proper personal protective equipment.

Processing the scene of a clandestine lab is the proverbial "accident waiting to happen." However, knowing the causes of accidents can help prevent and eliminate them. The major causes of accidents are lack of preparedness, inattention, carelessness, and fatigue.

There is no excuse for lack of preparedness. Investigators should have an idea of what manufacturing method the operator is utilizing prior to entry. With this knowledge, investigators should assemble the proper personnel and equipment to process the scene in a safe manner. The lab is not going anywhere; time is on the investigator's side. Therefore, the scene should not

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Table 2.2 Hazardous Materials Handling Guidelines

Hazard Type	Abatement
Explosive chemicals	**Do not handle chemicals** unless trained or absolutely necessary. The handling of chemicals should be left to trained personnel. Trained personnel can recognize chemical names that have explosive potential. They can also recognize chemical combinations or other situations with explosive potential. Untrained personnel should seal the area of the clandestine lab and wait for trained personnel to effect the abatement procedures. **Separate incompatible chemicals.** Trained personnel should separate chemicals with known incompatibilities. This will reduce the potential for these chemicals to accidentally combine and create an explosive situation. **Remove heat** from reaction mixtures. This will slow or stop the reaction taking place. This will reduce the amount of potentially explosive and toxic fumes that are being produced. Allowing the reaction mixture to reduce to room temperature naturally eliminates the effect of drastic changes in temperature. **Ventilate** confined spaces. This will reduce the concentration of explosive fumes in the area to below the explosive limit.
Flammable/ combustible	**Do not handle chemicals** unless trained or absolutely necessary. **Separate incompatible chemicals.** **Remove heat** from reaction mixtures. **Ventilate** confined spaces. **Isolate from ignition source.** All fires need fuel, oxygen, and a source of ignition. Removing the source of ignition removes the capacity to burn. (The muzzle flash from a discharged weapon is sufficient to ignite a flammable atmosphere.)
Acids and caustics	**Do not handle chemicals** unless trained or absolutely necessary. **Identify the pH** and acid type of all unknown liquids. Knowing the basic characteristics of the liquids will provide insight into how they should be segregated. (See Table 2.3 for bottle cap identification guide.) **Seal all containers.** This reduces the likelihood of the mixing of liquids if the containers are spilled. Also, some mineral acids produce fumes that can contaminate the air or mix with uncovered solutions containing incompatible mixtures. **Separate acids and caustics.** Segregating liquids by pH will prevent violent reactions if a chemical spill occurs. **Separate acids by type.** All acids are not created equal. Oxidizing mineral acids react violently when they come in contact with nonoxidizing mineral acids and organic acids. Segregating acids by type will prevent violent reactions if a chemical spill occurs.

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Table 2.2 Hazardous Materials Handling Guidelines (Continued)

Hazard Type	Abatement
Oxidizers and reducers	**Do not handle chemicals** unless trained or absolutely necessary. **Identify** known chemicals from container label. Unfortunately, there is no screening test for the rapid identification of oxidizers or reducers commonly used in the field by chemists investigating clandestine labs in the field. Unlabeled containers should be treated as unknowns, leaving the field identification to the chemical waste disposal company. **Seal all containers.** This reduces the likelihood of the mixing of incompatible chemicals if the containers are spilled. (*Cont.*)
Oxidizers and reducers (*Continued*)	**Separate oxidizers and reducers**. Segregating chemicals by class will prevent violent reactions if a chemical spill occurs. Segregate oxidizers from organic material and other combustible materials to prevent fire in case of a spill.
Compressed gas containers	**Do not handle containers** unless trained or absolutely necessary. **Do not handle containers unless absolutely necessary.** Safety outweighs the need to identify the contents of the container. **Do not open valves unless trained to do so.** **Close valves** of containers connected to reaction apparatuses. **Secure container** for chemical disposal company disposal.

Table 2.3 Acid Cap Color Code

Cap Color	Acid
Blue	Hydrochloric (HCl)
Yellow	Sulfuric (H_2SO_4)
Brown	Acetic (CH_3COOH)
Red	Nitric (HNO_3)
Clear/white	Phosphoric (H_3PO_4)
Black	Perchloric ($HClO_4$)
Black	Hydriodic (HI)

be entered or processed if the proper personnel and equipment are not present, unless exigent circumstances exist.

Inattention and carelessness go hand in hand. Many seasoned clandestine lab investigators routinely see the same manufacturing process and become lax in their handling of the situation. They begin to ignore common safety practices or fail to see obvious hazardous situations.

Fatigue can lead to such carelessness and inattention. Many clandestine labs are processed late at night, after a long protracted investigation or surveillance. Lack of sleep leads people to be not only tired but also frustrated. The lengthy procedure of safely processing a clandestine lab scene adds to

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the fatigue. Sleep deprivation also leads to many poor or hasty decisions, which in turn, can lead to undesirable consequences.

Thermal hazards are another type of physical hazard. In this context, they relate to the environmental temperature rather than to the heat of the reaction equipment in the lab, encompassing both extremes of hot and cold.

Heat stress occurs when the body is exposed to excess heat for an extended period of time. Such stress can affect the body's ability to regulate its temperature as well as other functions. Heat exhaustion can be debilitating but leaves no permanent effects. However, heat stroke can be fatal, and medical attention is required as soon as possible.

Figure 2.10 Group in Level B protection.

Heat effects can be minimized by acclimatizing the body to the temperature in the lab area prior to beginning work. It is not wise to go from an air-conditioned building or car to immediately working in an outdoor lab with an ambient temperature of 110°C. Allowing the body to get used to the temperature in the work area reduces shock to the body.

Hydrating your body with fluids containing electrolytes is a good preventative measure. Avoid drinks containing diuretics, such as caffeine (coffee, tea, soda). These cause the body to lose fluid faster than normal. These fluids could be used to cool the body, in the form of perspiration, if they are not eliminated through the urinary tract so quickly.

The personal protective equipment (PPE) used by investigators to process clandestine labs accentuates the problems related to heat (Figure 2.10). The characteristics of the equipment that protect the body from environmental hazards in the lab area work against the body's natural ability to cool. Such clothing does not "breathe" or allow the body's perspiration to evaporate, and thus cool itself naturally.

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Two common signs of heat stress are the loss of rational thought and the slowing of bodily functions. Inattention and carelessness lead to accidents. The reaction times of people suffering from heat stress slow noticeably. And, those with heat stress are easily recognizable (by someone else) by changes in body language, speech patterns, and perceptions of time.

It is imperative that the buddy system be used when working in situations involving elevated ambient temperatures. The buddy needs to be constantly aware of the condition of his partner. As soon as he sees signs of heat stress in his partner, they should both leave the area for a period of rehydration and cooling off.

The effects of heat can be minimized. Establishing a work–rest pattern will help reduce heat effects. Consistent breaks to cool the body and replenish fluid levels are good preventative measures in this battle. It is recommended that cotton underclothing be worn to wick the perspiration away from the skin. Also, during rest breaks, the PPE should be opened, allowing the body to breathe and cool.

Cold is the other side of the thermal hazard coin. As with heat, excess cold tends to distract a person's attention from the task at hand and lead to accidents. Cold stress occurs in cold, wet, and windy environments, with frostbite the most common injury. Hypothermia is the extreme case, which can result in unconsciousness or death if not addressed immediately.

To reduce the effects of cold, clandestine lab responders should dress appropriately for the weather conditions. Layers of warm clothing are a wise choice, because layers can be added or removed as needed. Avoiding windy and wet conditions, when possible, also reduces the effects of the cold. Staying physically active generates heat and keeps the body warm. However, excessive sweating should be avoided. When the activity stops, the excess perspiration evaporates, increasing the effects of the cold.

As with hot conditions, an established work–rest pattern reduces the effects of cold. Frequent rest breaks remove personnel from the cold environment and allow them to warm up. This also keeps them from generating an excess of perspiration that will lead to excessive cooling and gives them the ability to adjust the number of warming layers to the current environmental conditions.

Electricity is taken for granted in modern America. We plug something into an outlet, turn the switch, and it works. Electricity in the world of the clandestine lab operator is not that simple. The wiring of some of the equipment is makeshift, or the original design was unsafely altered (Figure 2.11). Electrical boxes that were originally wired to the local building code were altered to clandestinely divert current. A single outlet designed to draw 15 amps of current may have multiple extension cords attached to it, leading to equipment drawing 50 amps of current. Bare wires present a constant

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threat of electric shock or source of spark that can ignite a flammable atmosphere.

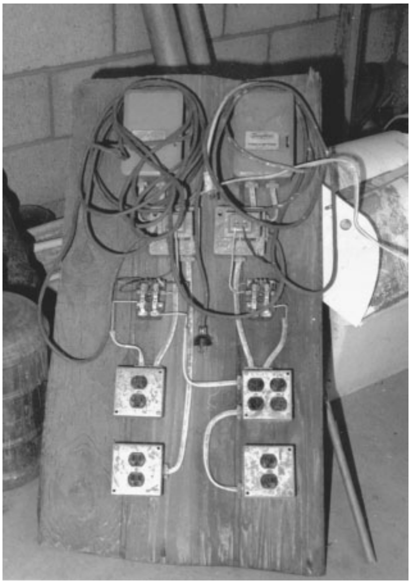

Figure 2.11 Clandestine lab wiring panel and wires.

To eliminate all electrical hazards prior to processing, turning off the electrical power to the site is simple but effective. However, prior to turning off the power, the assessment team needs to determine if there is some portion of the operation that would be adversely affected if the power were off. For example, it would not be wise to turn off the power if there was a cooling operation being powered by electricity. If the power were turned off, the cooling operation would stop, and potentially, the reaction could overheat, leading to an explosion or the like.

Confined spaces have limited entry and exit openings, unfavorable ventilation, and are not intended for continuous occupancy. Almost by definition, a clandestine lab is a confined space. To avoid detection, they are squirreled away in bathrooms, attics, closets, a workroom off a garage, or in small outbuildings. Ventilation is usually minimal or nonexistent. Even though it does not appear to be suitable for continuous occupation, some operators will spend hours or days at a time in the lab, while the manufacturing process is under way. It is common for the lab area to contain so much stuff that there is hardly any room to move. This increases slip, trip, and fall hazards.

When processing clandestine labs in confined spaces, the number of people in the area at one time should be kept to a minimum. Increasing the

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number of people in the area increases the potential for accidents and exposure to chemical hazards.

Personnel must be constantly aware of the limitations placed on them by their PPE. A person who normally has the agility of a rabbit is converted to a turtle when the PPE is worn. The PPE also reduces the person's dexterity and limits mobility. The use of a self-contained breathing apparatus (SCBA) increases the operating size of a person by placing an air tank (adding 12 additional inches) behind them. Air-purifying respirators (APRs) and SCBAs narrow peripheral vision and reduce the ability to verbally communicate. Unfortunately, these are necessary evils when working in an environment containing numerous unknown and potentially lethal hazards.

2.3 Hazard Abatement

A clandestine lab has a mixture of physical hazards and chemicals with varying hazard levels and chemical compatibilities. The hazards associated with some of the reaction by-products may be greater than those of any known chemicals. The following generic rules can be used when dealing with the hazards at a clandestine lab site:

Do not process a clandestine lab without the proper training. Training provides the knowledge with which to recognize the potential hazards involved in any scenario. It also provides the knowledge of how to abate the hazards when encountered. Processing a clandestine lab without the proper training can lead to serious injury of the individual and the surrounding personnel.

Do not process unless all personnel are present. The processing of a clandestine lab scene involves a team of people and incorporates many specialized tasks. This includes support personnel, such as the fire department and emergency medical services. Safety may be compromised if the processing commences without an individual with a specific expertise. The operation is not going anywhere. Unless there are exigent circumstances, safety concerns overrule expedience.

Do not process without the appropriate safety equipment. The chemical and physical hazards involved in clandestine labs pose a variety of health hazards to the personnel processing the operation. Chemicals can enter the body many ways, and specialized equipment is required to minimize such hazards. The use of the proper safety equipment will provide the responders with the protection necessary to minimize such effects of exposure. The operation should not be processed unless the proper safety equipment is present. As with the need for

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the proper personnel, the operation is not going anywhere. Therefore, for safety reasons, the operation should not be processed until the proper PPE is present.

Ventilate confined spaces. This reduces the concentration of explosive, flammable, or toxic fumes in the area to below the hazardous limit. Proper ventilation also provides an atmosphere in which the need for respiratory protection is reduced or eliminated. This, in turn, reduces the potential for accidents that result from the use of bulky PPE.

Isolate from the ignition source. All fires need fuel, oxygen, and a source of ignition. Removing the source of ignition removes the capacity to burn. Turning on a light or lighting a cigarette at an inappropriate time can serve as ignition sources. Enforcement personnel should be reminded that the muzzle flash from a discharged weapon is sufficient to ignite a flammable atmosphere.

Remove heat from reaction mixtures. This will slow or stop the reaction taking place, reducing the amount of potentially explosive, flammable, and toxic fumes being produced. Allowing the temperature of the reaction mixture to reduce to room temperature naturally eliminates the effect of drastic changes in temperature. This task should only be done by a trained chemist who is able to identify the type of reaction being utilized.

Identify known chemicals from container labels. Untrained personnel who encounter chemicals should relay any label information to trained personnel. Label information may provide trained personnel insight as to what chemicals are potentially at the location. The information could help establish potential manufacturing methods and additional chemicals that may be at the location. It is important to remember that not all labels accurately indicate the contents of their containers; however, it is a start in the flow of information.

Do not handle chemicals unless trained. The handling of chemicals should be left to trained personnel. They can recognize chemical names that have hazardous potential. They can also recognize chemical combinations or other situations with hazardous potential. Untrained personnel should seal the area of the clandestine lab and wait for trained personnel to begin the abatement procedures.

Do not handle containers unless absolutely necessary. Safety outweighs the need to identify the contents of the container. When practical, law enforcement personnel should take evidentiary samples of containers where they are located and allow hazardous waste disposal specialists to move such containers at the time of disposal. Every time a container is handled, there is potential for an accident. Therefore, reducing the

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number of times hazardous materials are handled reduces the potential for an accident.

Only trained personnel should do field testing. Field testing involves conducting chemical reactions with unknown chemicals. Trained chemists and hazardous materials responders have the training and experience to determine what field tests are appropriate for a given unknown. Applying an inappropriate field test may lead to an incompatible chemical reaction that creates a more hazardous situation than existed prior to the field test.

Powdered sodium cyanide can easily be mistaken for a controlled substance. Administering controlled substance field tests that are commonly available to law enforcement officers can prove to be a lethal decision. Many of these tests contain an acid that, when reacted with sodium cyanide, produces hydrogen cyanide. Exposure to hydrogen cyanide is extremely hazardous, if not deadly.

Beyond establishing the pH of a liquid and whether it is organic or aqueous, there are few field tests that will establish probative information for the clandestine lab investigator. Evidentiary testing should be restricted to laboratory conditions. However, field tests to establish potentially hazardous characteristics of chemicals may be done by trained personnel for the purpose of separating and segregating chemicals before the chemical disposal company arrives.

Separate and segregate incompatible chemicals. Trained personnel should separate and segregate chemicals with known incompatibilities. This reduces the potential for these chemicals to accidentally combine and create a more hazardous situation. Trained personnel use known label information and their training and experience to approximate the contents of unlabeled containers and separate them accordingly. They then segregate chemicals by placing those with similar properties in distinct groups. Placing a physical barrier between chemicals with known incompatibilities reduces the likelihood of chemical interaction if a spill should occur. Acids and caustics are differentiated by pH and are segregated. Acids are also separated by type, because all acids are not created equal. Oxidizers are separated from organic material and reducers. Listed in Appendix E are groups of incompatible chemicals that can be used to assist in the segregation process.

Seal containers. Sealing containers reduces the likelihood of incompatible chemicals combining. Some mineral acids produce fumes that can contaminate the air or mix with uncovered solutions containing incompatible mixtures. However, hot reaction vessels should not be sealed until they reach room temperature. If they are sealed prior to

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cooling, the act of cooling will create a vacuum seal, making the removal of the lid difficult and hazardous.

Do not handle compressed gas containers unless absolutely necessary. The contents of compressed gas containers are unknown. There is no way to safely determine such contents or the structural integrity of the container. The valves of compressed gas containers should not be manipulated unless absolutely necessary, because they could break off from corrosion. They may release an unknown toxic gas into the atmosphere. Close valves of containers only if they are connected to reaction apparatus. Compressed gas containers should be secured and left for the chemical disposal company to handle.

There are a number of subsequent steps clandestine lab responders can take to minimize the effects of physical hazards:

Be in good physical shape. The human body has the remarkable ability to compensate for a wide variety of physical stresses that are placed upon it. Proper exercise and diet maximize the body's defenses against physical stress. It is recommended that clandestine lab responders exercise regularly and eat properly. It is also wise to have yearly physicals to help determine any medical conditions that may have an adverse effect upon the body when placed under the physical stress involved in clandestine lab response.

Be rested. Being well rested also reduces the effects of mental and physical fatigue that lead to inattention and carelessness. This may not be realistic under the circumstances involved in the seizure of a clandestine lab. However, every effort should be made to minimize the effects of fatigue. Frequent rest breaks are necessary to reduce the effects of heat and cold stress on the body. These breaks also provide a mental break to reduce the effects of mental fatigue.

Drink liquids. Proper hydration is necessary to allow the body to regulate its internal temperature. It is wise for a responder to hydrate his body with liquids high in electrolytes prior to arrival at a clandestine lab scene. Replacing fluids during regularly scheduled rest breaks is essential to maintain the fluid levels the body requires for optimum efficiency. This suggestion cannot be emphasized too often, especially in hot climates. Avoid caffeinated drinks because of their diuretic effects.

Minimize number of people exposed. Keeping the number of people to a minimum in the lab area at any given time helps to reduce the potential for accident. A clandestine lab has a limited amount of space in which to operate. Increasing the number of people in this small space coupled with the reduction in agility and dexterity brought on by the limita-

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tions inflicted by the PPEs increases the potential for an accident. Also, by limiting the number of people in the lab area, it is possible to limit the number of people exposed to the maximum amount of hazard. Bottom line: if you do not need to be in the lab area, do not go in.

Utilize a buddy system. The buddy system is an essential safety consideration. The effects of heat and cold stress and chemical exposure may not be apparent to the person exposed. Exposure may lead to clouded thinking, irrational responses, distortion of time, and compromised motor skills. A buddy is needed to monitor the physical well-being of his partner and determine if he is functioning normally. At the first sign of abnormal behavior, it is the responsibility of the buddy to decide to leave the area as a pair for rehabilitation. Also, if one of the partners is involved in an accident, the buddy is there to render aid.

2.4 Summary

There are numerous hazards associated with clandestine laboratories, all of which can be minimized with proper training and education. The key is for the responders to use knowledge, skills, and abilities provided in the training to recognize potential hazards and to take the preventative measures necessary to minimize the potential for undesirable effects.

All clandestine labs have three things in common. The operators have little training, they are generally makeshift operations, and no two operations are exactly alike. Keeping these three principles in mind allows the scene investigator to have a greater appreciation for the number of things that can potentially go wrong while processing the scene of the operation.

While on location, all personnel should continually be evaluating the scene for potential hazards. What can explode? Is there anything or a situation that could cause a fire? Are firearms available to undesirable people? Are there unseen things or situations that could present a hazardous situation, either now or in the future? Finally, what can be done to reduce or eliminate these hazardous situations?

There are numerous things that can go and do go wrong at clandestine lab scenes. The post-scene debriefings of incidents in which something went wrong revealed a common thread. The undesirable effect was a direct result of someone not following the established safety procedures. The lesson being, using the established safety procedures and equipment reduces the effects of the hazards involved with clandestine labs to the degree that will allow anyone processing the scene to leave as healthy as they arrived.

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3 Basic Toxicology

Exposure to hazardous chemicals is one of the dangers of processing clandestine lab scenes. As described in the last chapter, the hazardous effects among chemicals vary widely. The short-term effects of a chemical exposure can differ from the long-term effects. In some instances, a single exposure can be lethal. In other situations, the effects are seen in the exposed person's offspring. That is why chemical exposure is the "silent" hazard.

Toxicology is the study of the adverse health effects of exposure to a chemical substance. As in many situations, knowledge is power. To have the power to protect oneself from the effects of chemical exposure, a person needs to know where the exposures can occur, how toxins enter the body, and how the various chemical classes will affect the body physiologically. Knowing how a chemical affects the body provides the knowledge required to protect against its toxic effects.

The goal in this chapter is to provide basic knowledge concerning toxic effects of hazardous chemicals encountered at clandestine lab scenes. Addressed in this chapter will be the entry routes by which chemicals enter the body, conditions that affect the absorption of chemicals, chemical toxicity ratings, types of toxins, and their systemic effects. This chapter is not a scientific treatise concerning the toxicology of hazardous material. It presents basic explanations of how toxic materials can enter the body and what happens when they do.

3.1 Entry Routes

Hazardous chemicals must have contact with the body to have a toxic effect. The point of contact determines the entry route and can affect the body's response to a particular toxin. For example, the body's response to a dermal exposure to sodium cyanide differs from ingestion of the same compound.

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Cyanide is a chemical asphyxiant that inhibits the blood's ability to carry oxygen. If the cyanide does not enter the circulatory system, i.e., through skin contact (dermal exposure), it cannot affect the blood's oxygen-carrying ability. However, if the sodium cyanide is ingested or inhaled, it can be adsorbed into the circulatory system, resulting in its toxic effects.

The three entry routes by which toxic materials enter the body are through inhalation, dermal adsorption, oral ingestion, and direct contact. As chemicals can have multiple hazardous effects (i.e., nitric acid is a corrosive, oxidizer, and poison), they can also have multiple toxic effects. The entry route of a toxin plays a role in what toxic effect the substance has on the body.

3.1.1 Inhalation

Inhalation is the most common and efficient entry route by which toxins enter the body. It is the most common entry route, because people have to constantly breathe to survive. (People can survive for extended periods without eating or touching things.) Thus, the respiratory system (Figure 3.1) is constantly exposed to the outside environment, which may contain toxic substances. It is the most efficient entry route, because the respiratory system provides a direct conduit from the environment to the body's circulatory system. Once the toxin is in the circulatory system, it is a simple ride on the blood express to the toxin's target organ of choice.

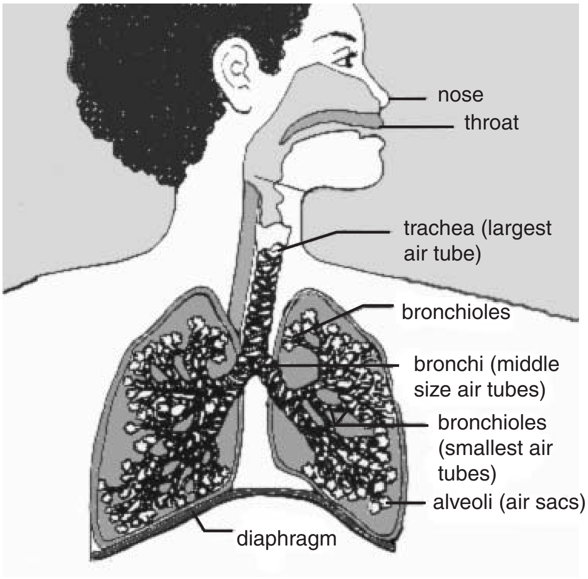

Figure 3.1 Respiratory system.

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The respiratory tract is comprised of the upper airway, the lower airway, and the alveoli. Each portion has a specific function. Portions filter out toxic material that would inhibit the oxygen–carbon dioxide exchange in other portions. Other portions exchange toxic materials between the blood and the air that is breathed.

The upper airway is comprised of the nose and larynx. Its purpose is to increase the relative humidity of the incoming air. It is lined with hairs called ciliated epithelia that keep large particles from the incoming air from getting into the lower airway and alveoli. The upper airway's moist environment provides an atmosphere in which water-soluble materials can dissolve and potentially enter the circulatory system.

The lower airway is comprised of the trachea, bronchi, and bronchioles. They are lined with mucus-coated ciliated epithelia. The purpose of the lower airway is to prevent particles that passed through the upper airway from getting into the alveoli. The ciliated epithelia move the trapped particle up the lower airway to the oral cavity for elimination. Smoking and the use of cough suppressants can, in effect, paralyze the ciliated epithelia, affecting their ability to filter and remove particles from the incoming air. Water-soluble toxins can be adsorbed in the mucus, also affecting the function of the ciliated epithelia.

The alveoli are the tiny sacks attached to the bronchioles. They contain the blood vessels that exchange oxygen and carbon dioxide. Another function of the alveoli is to exchange volatile toxins with the incoming air and eliminate them in the expired air.

The alveoli provide the largest surface area of the body that is constantly exposed to the environment. They provide a direct connection between the environment and the body's circulatory system. Toxins can enter the circulatory system directly at this point. The act of breathing constantly refreshes the supply of toxic substances that can be introduced to the circulatory system for distribution throughout the body.

Chemicals do not absorb into the body at the same rate. A person's physiology and the chemical properties of the toxin work in tandem to affect how readily the toxins are absorbed through the respiratory system. Respiratory rate, particle size, and chemical solubility are the principle issues that affect how readily a toxin is absorbed by the body.

The exposure rate to a toxin is directly proportional to the amount of air breathed. The faster the breathing rate is, the greater the amount of air that enters the lungs. This leads to an increase in the amount of toxic exposure. The slower the breathing rate is, the lower the amount of air that enters the lungs. This leads to a decrease in the amount of toxins that can enter the respiratory system for absorption.

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To limit toxic inhalation exposure, it is recommended that the respiratory rate be kept to a minimum by being in good physical condition and maintaining composure during processing activities. Being in good physical shape influences the efficiency of the lungs' oxygen exchange ability, which assists in reducing the respiratory rate. Being in an excited state increases the respiratory rate. Maintaining composure keeps the respiratory rate low, minimizing the exposure.

The substance's particle size affects if or where the toxic effect will result. Solid particles ranging in size from 5 to 30 μm are filtered out in the upper airway. Particles in the 1 to 5 μm range are filtered out in the lower airway. They collect on the mucus-covered epithelium and are transported to the oral cavity for elimination. These particles can also embed themselves into a portion of the respiratory system, causing infection or disease. Asbestos is an example of an insoluble particle that embeds itself into the respiratory system, producing adverse effects.

When working in toxic environments, it is imperative that responders do nothing to affect the ciliated epithelia's ability to remove particles from the incoming air. Smoking and the use of cough suppressants could inhibit the cleansing movement of the cilia, allowing particles to embed themselves into some portion of the respiratory system or to be introduced into the circulatory system.

A substance's water solubility will determine its toxic effects through inhalation. Water-soluble substances will dissolve in the mucus of the upper and lower airways. This may produce a localized toxic effect or allow the compound to be absorbed in the circulatory system. Water-insoluble substances can travel past the upper and lower airways into the alveoli, where they can enter the circulatory system.

3.1.2 Dermal Absorption

Dermal absorption is the second route of entry by which toxins enter the body. Many people have the misconception that the skin is impervious to everything. If contact with the substance does not produce some sensation of pain or discomfort, there must be no toxic or hazardous effect. This is not the case when considering the skin as an entry route for toxic materials.

The skin is comprised of the dermis and epidermis (Figure 3.2). By weight, it is the largest organ of the body. The skin has two main functions. First, it protects the internal organs of the body from adverse environmental conditions. Second, it regulates the body's temperature. Both of these functions affect how toxic substances are absorbed into the body.

The epidermis is the top layer of skin cells that provides the first line of defense from environmental toxins. It can intrinsically repel the toxic effects

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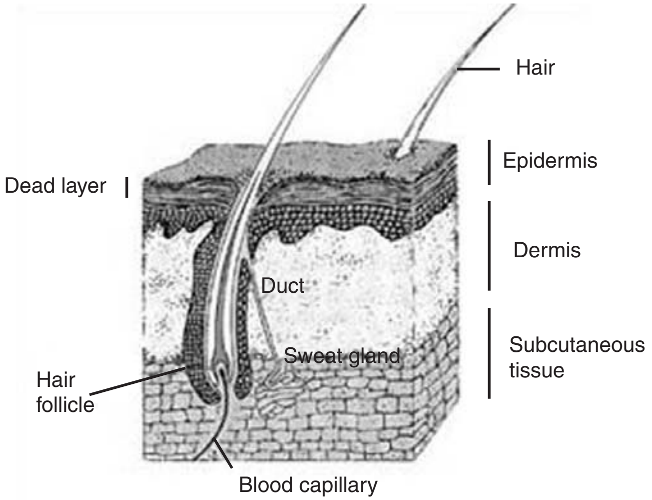

Figure 3.2 Skin cross-section.

of a number of substances. The condition of the epidermis plays a critical role in its ability to repel toxins. Dry skin or skin damage as a result of cuts or abrasions can result in toxins bypassing the protective traits of the epidermis. Corrosives can damage the skin and provide an opening for other toxins to enter.

The dermis comprises the lower layers of the skin. It contains the sweat glands and ducts, oil glands, fatty cells, connective tissues, and blood vessels. It provides a nonselective diffusive environment by which toxins travel into the circulatory system.

The rate toxins are absorbed through the skin is affected by a number of factors. The relative effect of one may affect the relative effect of one or more of the others when combined. The factors that affect the rate of dermal absorption include skin damage, hydration state, temperature, concentration, and carriers. Each factor affects whether a substance is repelled or absorbed by the skin.

Skin damage is the simplest to understand. The skin can be equated to the plastic coating that protects a package's contents from water damage. A cut or an abrasion in the skin can be equated to a break in the package's plastic coating. Breaking the plastic coating provides an entrance for water to enter the package. Breaking the protective coating provided by the epidermis provides toxins a direct route into the circulatory system.

The hydration state of the epidermis can affect its ability to absorb or repel toxic substances. The epidermis has an optimum hydration state. If it is too dry, it may more readily adsorb liquid toxins or let them pass through to the dermis. If the epidermis is overhydrated, it may provide an environ-

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ment that will allow the toxins to diffuse through it into the blood vessels in the dermis.

The ambient temperature affects dermal defenses in a couple of ways. It can affect the hydration state of the skin. Skin perspires in elevated temperatures. Perspiration increases the hydration state of the epidermis, leading to the effects described above. The sweat ducts also provide a conduit for toxins to travel past the epidermis into the dermis and potentially into the circulatory system.

A second effect concerns the blood vessels in the dermis. In hot environments, the blood vessels expand to increase the blood flow in the dermis in an effort to reduce the body's internal temperature. In cold environments, the blood vessels in the dermis contract to restrict the blood flow in the dermis in an effort to keep the body warm. In elevated temperatures, the surface area of the blood vessels increases, increasing the ability for a diffusion transfer through the dermis into the circulatory system.

The concentration of a substance plays a role in how it affects the skin. The epidermis is resilient. It can compensate for the toxic effects of low concentrations of a given substance. Exposures to these low concentrations produce little or no toxic effect. However, higher concentrations of the same substance can produce devastating effects.

Chemical carriers can provide a ride for a toxin through the skin's natural defenses. A solid toxin that the epidermis would normally repel may pass through it, if it is dissolved into a solvent carrier that permeates the epidermis. The solvent shuttles the dissolved toxic substance past the epidermis into the dermis and then into the circulatory system.

3.1.3 Ingestion

Ingestion is the final mode by which toxins enter the body. This is the least effective way for toxins to enter the system. The physical state of the substances introduced through this mode, and the nature of the digestive tract, keep the amount of toxin and toxic effects to a minimum (Figure 3.3).

Solids and liquid substances enter the body through this mode, which makes intentional ingestions difficult. The mouth is a relatively guarded entry point. People have the common sense to not intentionally eat or drink something that has hazardous potential, as opposed to dermal contact, with which unintentional direct contact with the substance is common.

Inadvertent ingestions of toxic substances can happen. People in toxic environments inadvertently handle items that are placed in the mouth. Smoking and eating in an area where toxic substances are present affords the opportunity for toxins to be placed in the mouth if a contaminated hand handles the cigarette or food. For example, people taking notes at a clandes-

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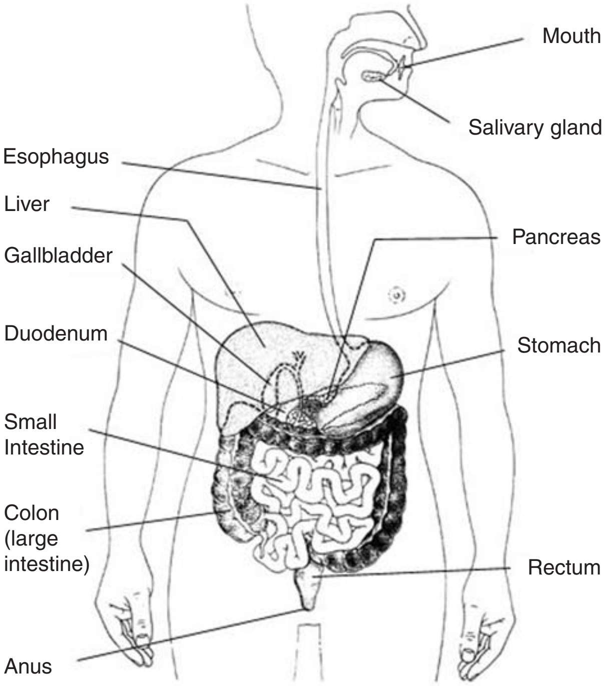

Figure 3.3 Gastrointestinal tract.

tine lab scene will place the pen with which they have been writing in their mouths. Just before, they handled toxic chemicals that have now been ingested into the gastrointestinal tract. The only saving grace in this incident is that the concentration of the substance may be low enough that it will not produce a toxic effect.

A third consideration is the environment within the digestive system. The highly acidic and alkaline environments in the various portions of the gastrointestinal tract have a tendency to neutralize the toxic effects of many substances before they can be introduced into the bloodstream. Even if a toxin is introduced, interaction with the digestive juices may alter the toxic properties of the substance before it has a chance to enter the circulatory system.

3.2 Modes of Action

Every substance has a different effect on the body. The mode of action refers to the physiological system, which is affected by the exposure. The three general modes of action consist of the physical, chemical, and enzymatic.

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The physical mode of action refers to how the substance interacts with all tissues of the body. The substance's hazardous characteristics can be used to characterize its physical mode of action. For example, a substance with corrosive properties will produce the same effect, no matter the tissue with which it comes into contact, i.e., corrosive substances are nonselective in the type of tissue with which it reacts.

The chemical mode of action refers to how a substance interacts with specific tissues of the body. It can be characterized by generic toxic properties of the substance. For example, chemical asphyxiants interfere with the blood's ability to carry oxygen. The reactions are tissue specific and deal with the chemistry between the substance and tissue involved.

The enzymatic mode is also referred to as the physiological mode. It refers to how the substance interacts with specific enzymes in the body. The substance can enhance or inhibit the processes of the enzyme it affects.

3.3 Influences on Toxicity

A number of variables will affect the body's response to a given toxin. The length and the degree of exposure play a determining role. The chemical's physical properties, its concentration, and the duration of exposure have their own influences on the body's reaction. Even environmental factors, such as the temperature, play a role in how a substance will react with the body. The variables that affect the toxicity of a substance are the length of the exposure, the degree of exposure, toxicity factors of the substance, factors concerning the exposure, environmental factors, and factors concerning the person exposed.

3.3.1 Length of Exposure

As discussed in Chapter 2, the length of exposure to a hazardous substance will determine the effects the substance has on the system. Also, the short-term effects differ from the long-term effects.

Exposures to toxic hazards have acute and chronic effects. Acute effects are experienced with exposure to hazards of high concentrations of short duration. The effects are felt immediately. The exposed will recover if the exposure does not exceed the lethal limits. Chronic effects are generally experienced with exposure to hazards of low concentrations over a long period of time. The effects are cumulative, creating a toxic effect. These effects are usually different than the acute effects of the same hazard.

The duration of an exposure may be too short to produce an acute effect. The body may have enough natural defenses to ward off any adverse effects. However, because of the depletion of these natural defenses, subsequent

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short-term exposures may have a compounded acute effect if the body has not had an opportunity to regenerate its defense mechanisms.

In some instances, the body stores the substance in the fat cells, liver, or other target organ. The concentration increases until it reaches a toxic level. Numerous insignificant short-term exposures may not produce any acute effects. However, the cumulative effect may produce a chronic toxic effect.

3.3.2 Degree of Exposure

The degree of exposure differs from the length of exposure. Exposure length is strictly a measure of the time a person is exposed to the substance. The degree of exposure relates to the substance's chemical properties, the concentration, and the specific duration of the exposure. These properties are interrelated and affect how the substance will react with the body. Degree and length of exposure are interrelated in that by changing one of the properties of the degree of exposure, the length of time required to produce a toxic effect will be altered. The group of exposure properties interacts in different ways. The effect produced results from the relationships between the different factors surrounding the exposure. The factors concerning the compound, the circumstances surrounding the exposure, the exposed person, and the environment of the exposure will combine and establish the total hazardous potential of the exposure.

3.3.2.1 Compound Factors

The substance has properties that establish its inherent toxicity. Its chemical properties, concentration, duration of the exposure, and interaction with other chemicals will interact in different ways to produce different effects. The ratio of these is different in each exposure. Thus, the effects of the same substance will vary depending on this ratio.

Each chemical has an inherent set of physical and chemical properties. Some have a hazardous potential. Some physical properties prevent the substance from interacting with the body. For example, under normal circumstances, solids and liquids cannot be inhaled. However, change the solid into an airborne dust or a liquid into an aerosol, and these physical states can be inhaled.

The substance's chemical properties will determine what effects it will have on the body when an exposure occurs. For example, under normal circumstances and without outside influences, a corrosive chemical will not burn or explode. However, contact with an incompatible property may create an explosive situation.

The concentration of the substance during the exposure will affect the body's response. The body has the ability to compensate for exposure to low concentrations of a wide variety of chemicals. Its internal defenses can neu-

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tralize or compensate for the toxic effects of a given substance. However, high concentrations of the same substance during the same exposure time may overload the body's ability to defend or compensate for the toxic effects.

The duration of the exposure will affect how the body reacts. The effects of instantaneous contacts differ from those of prolonged contacts. Instantaneous contacts may not allow enough time for the substance to interact with the body, or the body may have sufficient defenses to compensate for that exposure duration.

A dramatic example of this is exposure to liquid nitrogen. Liquid nitrogen has a boiling point of –196°C (–321°F). The human body can tolerate a direct instantaneous exposure to liquid nitrogen. Beyond that, the temperature of the liquid nitrogen overcomes the body's ability to compensate, and severe damage results.

Interaction with other substances can potentially affect the substance's toxicity. Contact with other chemicals may alter the substance's physical state, which may affect the entry routes available to the substance. For example, the reaction between cyanide salts and acids converts cyanide salt from a solid (with low inhalation potential) to hydrogen cyanide gas (with high inhalation potential). Dissolving a substance into a liquid may alter its ability to be adsorbed through the skin.

3.3.2.2 Exposure Factors

The circumstances surrounding the exposure will affect hazard potential. The entry route and the duration and number of exposures will determine what the body will experience. The combination and ratio of these factors will affect the body's toxic response.

The entry route is the primary factor in the exposure scenario. If the substance can neither get into nor have contact with the body, it cannot produce a toxic effect. As discussed earlier, different entry routes provide access to different physiological systems. A toxin's effect may differ depending on the entry route. For example, under normal conditions, cyanide salts have a negligible potential for dermal absorption. However, they are readily adsorbed through ingestion. A person can handle these highly poisonous substances with minimal effects from dermal absorption. However, when the person's contaminated hands come into contact with food that is then ingested, that insignificant exposure just became lethal.

The number of exposures influences the toxic effects of a substance. Certain substances are known as sensitizing agents. An initial encounter with the substance may not produce an effect. However, all subsequent exposures produce a toxic reaction. Other substances can have a cumulative effect. The body may store a substance until the concentration builds to the point where it reaches a toxic level.

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3.3.2.3 Personal Factors

Personal factors can be directly attributed to the exposed individual. The exposed person's age, sex, health, and genetics are directly related to how the person's body will react to an exposure to certain toxic substances.

A person's metabolism changes over time. The metabolism of an infant is different than that of a teenager entering puberty. A young adult's body's ability to bounce back from injury and disease is greater than that of someone who reached retirement age. This difference in metabolism between the ages dramatically affects the body's ability to fight the effects of toxic substances.

A person's sex may determine if a person will be affected by an exposure to a particular toxic substance. In some instances, the metabolism of males differs from that of females. In other instances, a toxin may target a specific organ. If the toxin specifically targets the reproductive system of a particular sex, it will not have a detrimental effect on the opposite sex. This can be of special concern to women of reproductive age. There is also a group of toxins that, when exposed to, can cause birth defects (teratogens) or fetal death (embryonic toxins).

The health condition of the person exposed to toxic substances will affect the body's response to the exposure. The body's ability to counter the effects of exposure to toxic substances is diminished if the immune system is fighting disease or infection. Poor health extends beyond illness. Fatigue can be included in this category. If the body is run down due to lack of sleep or other fatigue factors, its ability to ward off toxic effects is reduced, because the body's metabolism is not functioning at its optimum level for the person's age and normal physical health.

A person's genetic makeup will play a role in how certain toxic substances will affect the body. Some people are genetically predisposed to have a toxic reaction to a substance that another person is not affected by. Just as some people are allergic to dogs, pollen, or a variety of other allergens, some people will demonstrate a toxic reaction when exposed to a particular chemical, while others will exhibit no toxic symptoms to the same exposure.

Factors related to the environment affect if or how a toxin enters the body. The carrier, ambient conditions, and chemical interactions determine whether a particular exposure will produce toxic effects.

The carrier a toxin is associated with can provide the toxin an entry route that would not normally be available to it. Cyanide salts are an example of a solid poisonous substance that cannot easily enter the body under normal circumstances. However, accidental contact with ingested food provides the carrier the needed entry route. The same cyanide salt that cannot normally be absorbed dermally can enter the system through the skin if it is dissolved in the appropriate carrier solvent under the appropriate ambient conditions.

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The ambient conditions during the exposure also have their effects. Cold slows the body's metabolism to an extent. Cold affects dermal exposures by closing pores and restricting blood flow along the surface of the skin. Inversely, heat increases the potential for toxic exposures. Heat opens pores and increases the hydration state of skin, which increases the potential for solvent-soluble toxins to permeate the epidermis and enter the dermal layer, which contains blood vessels. Also, blood flow in the surface of the skin is increased in an effort to cool the body. Dilated capillaries provide a greater surface area and, therefore, greater potential for toxins to enter the bloodstream.

3.3.2.4 Distribution and Elimination

Many toxins do not stay at the initial contact site. They enter the body through one of the exposure routes and travel through the body to a specific target organ. Movement of the toxins through the body is facilitated by the circulatory system, which consists of blood and lymphatic systems. These systems also provide a method with which to remove toxins from the body.

In Figure 3.4, the flow of toxins is graphically depicted. Toxins enter the circulatory system via inhalation, dermal absorption, or ingestion. Liquid- and water-soluble solids are filtered out in the liver and kidneys and then are excreted from the body in urine. Solid toxins are excreted in feces. Volatile toxins are exchanged with incoming air and expelled in expired air. Other toxins are transported into cellular fluid, where they are stored in the organs, soft tissue, or fat cells.

3.4 Toxicity Measurements

There are a number of ways to establish toxicity of a substance. Some of the measurements relate to the lethal dose. Others have to do with the amount a person can be exposed to before they feel any effects. Other measurements relate to the instantaneous exposure or the amount of exposure a person can experience over a period of time. All of these measurements identify the dose threshold of a substance that a person can experience before suffering adverse effects.

Most toxicity measurements are derived from epidemiological and animal test data. The test values are usually derived from exposures given to rats that are then extrapolated to human ratios. Values are reported as ratios of weight of substance per weight of subject. For example, a toxic value of 5 mg/kg rat means that 5 mg of substance will produce the toxic effect in a 1 kg rat. This can be extrapolated to mean that 500 mg of substance would be needed to produce the same effect in a 100 kg human.

Many substances have an established LD_{50} value. The LD_{50} is the concentration of a substance that produces a lethal response in 50% of the test

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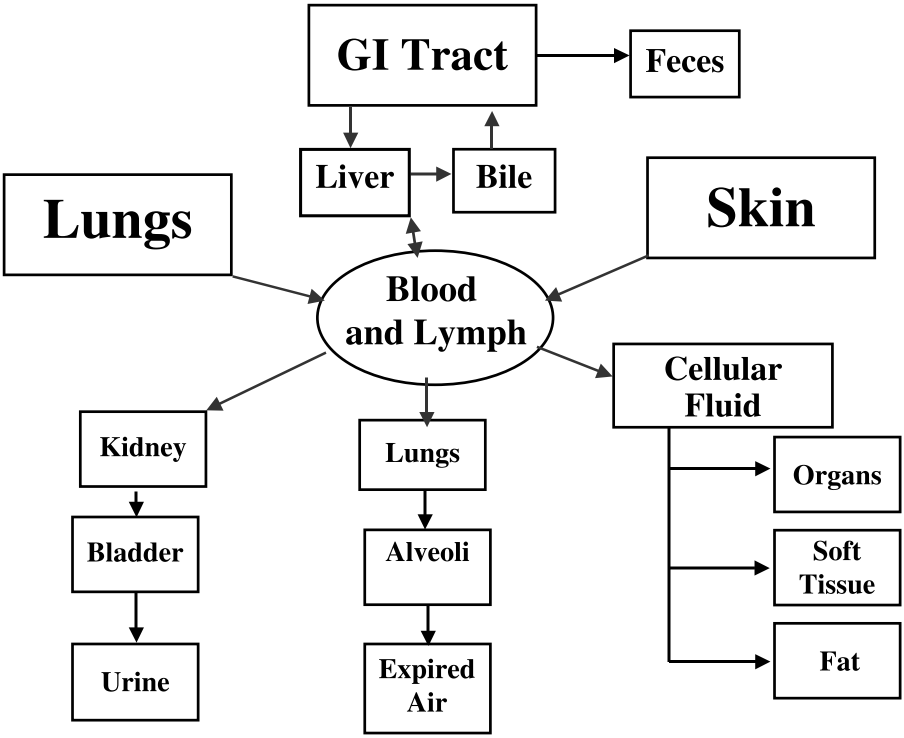

Figure 3.4 Toxin distribution chart.

population. In other words, it is the concentration of the substance that will kill 50 of 100 of the test subjects.

In the body, substances will react differently. Some have an immediate effect. Others can come into contact with the body at low concentrations without the body demonstrating symptoms of exposure.

Low-dose-response substances produce an almost immediate effect on the body (Figure 3.5). The percent of the population affected by exposure to a given substance increases with the concentration of the substance. The rate of increase of the affected population will vary between substances. At some point, the concentration will reach the LD_{50} for the substance. Eventually, a concentration will be reached at which everyone in the population will experience the toxic effects of the substance.

Substances with a high-dose response will not produce a toxic response until a certain concentration is reached (Figure 3.6). Up to that point, the body's natural defenses can counteract the toxic effects of the substance. Once the concentration of the substance reaches a threshold value, the population will begin to exhibit toxic effects. At that point, the percentage of the population experiencing toxic effects increases with dosage concentration.

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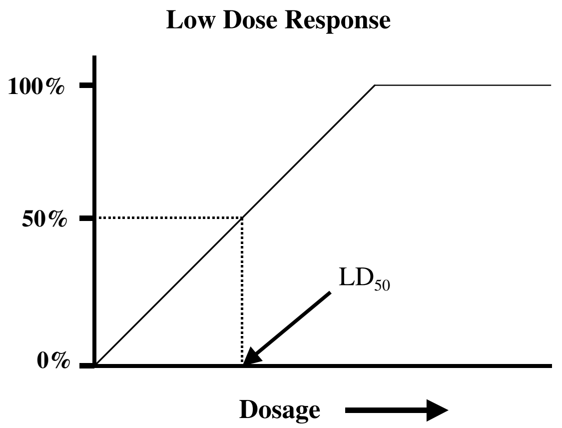

Figure 3.5 Low-dose curve.

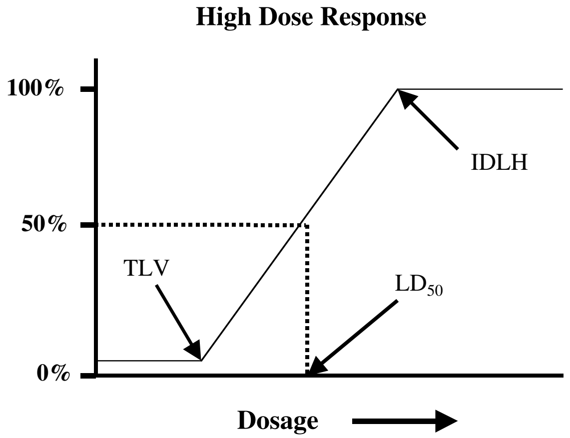

Figure 3.6 High-dose curve.

The relative toxicity of a substance is a function of the substance. Each compound has a relative toxicity. Terms commonly used to describe the toxicity of a substance are listed in Table 3.1. Each term has an associated concentration range. Extremely toxic compounds can cause death upon minute exposure, while you can literally bathe in relatively harmless substances before a toxic effect is experienced.

3.4.1 Exposure Guidelines

A number of agencies have established guidelines used to determine the toxic levels of various substances. The National Institute for Occupational Safety and Health (NIOSH), the Occupational Safety and Health Administration

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Table 3.1 Relative Toxicity

Rating	LD_{50} (Oral Rat)	Example
Extremely toxic	<1 mg/kg	Hydrogen cyanide (HCN)
Highly toxic	1–50 mg/kg	Mercuric chloride ($HgCl_2$)
Moderately toxic	50–500 mg/kg	Sodium hydroxide (NaOH)
Slightly toxic	0.5–5 gm/kg	Cyclohexanone
Practically nontoxic	5–15 gm/kg	Methanol
Relatively harmless	>15 gm/kg	Water

Source: Drug Enforcement Administration, Clandestine Laboratory Training Guide, Vol. 1, p. 22.

(OSHA), and the American Conference of Governmental Industrial Hygienists (ACGIH) have their own rating systems. Each measures a specific effect and is used to establish safety parameters with which to create a safe work environment.

The threshold limit value (TLV) is an exposure guideline established by the ACGIH. It corresponds to the concentration required to produce toxic symptoms. Low TLV values indicate a substance with a low-dose response. High TLV values indicate a substance with a high-dose response. The exposure concentration values are reported as either a time–weight average (TWA) or as a ceiling (c). The TWA value is the average concentration a person can be exposed to over a period of time (either 8 or 40 hours). The c value is the maximum concentration for an instantaneous exposure.

The relative exposure limit (REL) is the exposure limit established by the NIOSH. These values are similar to the TLV values and are directed toward industrial applications.

The most relevant exposure level to clandestine lab seizures is the IDLH. The IDLH is the concentration that NIOSH determined to be immediately dangerous to life and health. It is the concentration at which death or serious injury will be caused upon a single unprotected exposure.

The OSHA established permissible exposure limits (PEL) for numerous substances. These values are similar to the NIOSH REL values and can be found in Chapter 29 of the Code of Federal Regulations (CFR), Section 1910.1000, Table Z-1.

The significance of the PEL values is that they hold legal weight. An employee cannot be exposed to concentrations exceeding PEL of a substance without appropriate personal protective equipment (PPE). Law enforcement and emergency responders are not exempt from these regulations. Even under exigent circumstances, law enforcement and emergency responders are required to adhere to the PPE requirements established by 29 CFR 1910.120 when concentrations exceed the PEL.

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3.5 Toxin Properties

Now that we know how toxins enter and affect the body, we can discuss toxins. A toxic material is a substance that in relatively small quantities is capable of producing localized or systemic damage. Toxic materials come in a variety of physical states and can affect the body in a number of different ways.

3.5.1 Physical States

Toxins come in all physical states. They can be in the basic physical states of solid, liquid, or gas. They could be in some combination of these forms, such as fumes, smoke, aerosols, mists, vapors, or dust. Knowing the various physical states of a toxic substance that can be encountered provides a means for determining what types of PPE will be required.

All substances have a normal physical state of solid, liquid, or gas. The normal physical state is related to the standard temperature and pressure (uncontained at room temperature). Changing the temperature or the pressure conditions can affect the physical state of the substance.

Water is the simplest substance that can be used to demonstrate effects on temperature. Water is a liquid in an open container at room temperature (25°C). When the water's temperature is reduced to 0°C by placing it into a freezer or exposing it to liquid nitrogen, the water turns into a solid. When temperature is elevated to 100°C by adding heat from an external source, the water turns into a gas.

Changing the pressure conditions under which the substance is stored will affect the substance's physical state. Increasing the pressure in a closed container will condense a gas into a liquid, and the container will experience an increase in temperature. (That is why a bicycle tire's temperature increases as air is added.) Reducing the pressure over a liquid will convert the liquid into a gas and reduce the temperature of the container. (That is why an aerosol can gets cold when the contents are released.)

The conditions encountered in clandestine labs are dynamic. Ambient temperatures can be at extremes of hot and cold. Boiling reaction mixtures can vaporize substances that are normally liquids at room temperature. The excessive heat generated in an automobile in 110°F weather may pressurize chemical containers. Rapidly cooling a boiling liquid that was enclosed in a container may create a vacuum, which may make opening the container difficult. These environmental factors will affect the physical state of the substance.

The dynamics of the environment the substance encounters causes hybrid physical states. They are transitory or a combination of the basic physical states. These hybrid physical states include vapors, fumes, smoke, aerosols, mists, or dust.

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A vapor is the gas phase of a substance that is a solid or a liquid at standard temperature and pressure (room temperature). Vapors are produced when the temperature of the substance is elevated through natural (high ambient temperatures) or artificial means (heat added to a reaction mixture). The substance returns to its natural state when its temperature is reduced.

Fumes are vapors from substances that are solids at room temperature. They are the result of heating the substance, which produces airborne particles less than 0.1 μm in diameter. They can aggregate into fine clumps and eventually settle out of the air. Lead and iodine are examples of substances that can produce fumes.

Smoke is the result of incomplete combustion. The particle size is greater than 0.5 μm. These particles do not generally settle out of the air.

An aerosol is a stable suspension of solid or liquid particles of various sizes in the air that will eventually settle out of the air. Aerosols are usually the result of a mechanical distribution of the substance that atomizes the substance and disperses it into the air. The size and weight of the particles do not allow them to remain airborne.

Mists are liquid aerosols formed by liquid vapor condensing on airborne solid particles, which may or may not be visible.

3.5.2 Toxic Properties

Exposure to toxic materials produces localized or systemic damage to the body. That is, the toxic effect is experienced at the point of contact or when the toxin enters the body, travels to the target organ, and disrupts its function in some manner. Localized effects are experienced immediately. Systemic effects may be seen immediately, may not manifest for years, or may occur in the exposed person's offspring.

In Chapter 2, the hazardous properties of the chemicals found in clandestine labs were discussed. Discussed in this section will be how the hazardous properties correlate to toxic effects on the body. The toxic effects can be divided into corrosives, asphyxiants, irritants (respiratory, systemic, external), and special toxins.

Corrosives are localized toxins that can cause visible damage or irreversible alteration to human tissue at the point of contact. They include acids and caustics. The effects are generally considered to be localized. However, the damage can lead to systemic effects. For example, exposure to hydrochloric acid fumes can produce localized damage to the alveoli. This localized damage produces a systemic effect on the body's respiratory system.

Asphyxiants affect the supply of oxygen to the body. Even small reductions in the oxygen supply to the body can potentially produce a variety of effects on the body. Asphyxiants are divided into simple and chemical.

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Table 3.2 Oxygen Deficiency Effects

Percent Oxygen	Effects
21	No abnormal effects
12–16	Increased breath volume
	Accelerated heartbeat; impaired attention, thinking, and coordination
10–14	Faulty judgment and coordination; rapid onset of fatigue; intermittent respiration; permanent heart damage may occur
6–10	Nausea and vomiting; inability to perform vigorous movement or loss of all movement; unconsciousness and death
<6	Spastic breathing; convulsive movements; death in minutes

Source: Drug Enforcement Administration, Clandestine Laboratory Training Manuals, Vol. 1, p. 25.

Simple asphyxiants displace the oxygen in the atmosphere. There is no direct interaction with the body. They simply reduce the amount of an essential element available to the body. In Table 3.2, the effects that can be felt as the result of lack of oxygen in the air are presented. These chemicals may be inert when they come in direct contact with the body. Freon, which is used legitimately as a refrigerant and clandestinely as an extraction solvent, is an example of a simple asphyxiant.

Chemical asphyxiants are substances that affect the blood's ability to carry oxygen. This involves a chemical reaction with hemoglobin. The results will be similar to the oxygen concentration in the air being reduced. Carbon monoxide (CO) and hydrogen cyanide (HCN) are examples of chemical asphyxiants.

Irritants are compounds that disrupt the function of the system at the point of contact. They commonly affect the respiratory system. Water-soluble irritants such as acids can be absorbed into the mucus of the upper respiratory system, disrupting the ciliated epithelia's ability to function properly. These effects are usually experienced immediately. Non-water-soluble irritants travel into the lower respiratory system. The irritating effects disrupt the lung's ability to perform its oxygen exchange function. Asbestos is the most notorious non-water-soluble irritant. However, phosphine and phosgene gas, which are by-products of methamphetamine manufacturing operations, are examples relating to clandestine lab operations.

Allergens are substances that cause an immunological response upon exposure. The effect may be localized or systemic, and the point of contact determines the effects. For example, a dermal exposure to a substance may or may not produce a reaction. However, ingesting the substance may cause nausea, vomiting, or some other allergic reaction.

Systemic toxins affect the function of a physiological system. They travel from the point of contact to the target organ before the toxic effect is experienced. The central nervous system is affected by asphyxiants, hydrocarbons

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(such as hexane), and metals (like lead). Aromatic solvents, such as benzene and toluene, can affect the circulatory system. The function of the liver can be affected by aromatic compounds, chlorinated solvents (such as chloroform and carbon tetrachloride), and hydrocarbons. Halogenated compounds, such as Freon and metals, affect kidney function. The spleen is affected by halogenated aromatic compounds. Aromatics, pesticides, and organic metal compounds affect the reproductive systems. Listed in Appendix F are the target organs of the chemicals commonly associated with clandestine labs.

External toxins target the body's external organs. Various solvents, oils, metals, and corrosives target the skin and affect its ability defend itself from toxic exposures. The eyes are the external target organs for corrosives, solvents, oils, and lacrimators.

Special toxins are the "silent" toxins. Their effects are not seen immediately and may produce effects unrelated to the effects of the initial exposure. Carcinogens are substances that cause uncontrolled cell growth (cancer). Mutagens are substances that cause changes in the genetic code. Teratogens cause nonlethal congenital birth defects. The effects of mutagens and teratogens will be seen in the offspring of the exposed person. Embryonic toxins will cause fetal death. Reproductive toxins are sex specific and target the reproductive organs. Also listed in Appendix F are the toxic effects produced by exposure to the chemicals encountered in clandestine labs.

3.6 Summary

The chemicals encountered in clandestine labs can produce a variety of effects on the human body. Some are inert. Others generate an immediate lethal response. A great majority are somewhere in the middle.

The toxic effects of the chemicals involved in clandestine labs have the potential to affect the personnel processing the clandestine lab scene, including those beyond the personnel who have direct contact with the clandestine lab scene. There are many unwilling people who can potentially experience the toxic effects of the chemicals in a lab. The operators; the people living in the lab area, including spouses, their significant others, and children; and the people who subsequently move into the apartment, house, or motel room that was not properly decontaminated after the lab was removed, may potentially come in contact with the toxic materials. The responders may inadvertently bring toxic substances home or to the office, exposing families and coworkers to the hazards. The people in the criminal justice system, who come in contact with the evidence throughout the adjudication process, including people in the property room, court clerks, and attorneys, may also be exposed.

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As stated at the beginning of this chapter, knowledge is power. Knowledge provides the power to prevent toxic exposure. However, with knowledge of all of the potentially harmful things that can happen as a result of encountering any or all of the toxic substances, the clandestine lab investigator must ask the question: "Why am I investigating clandestine labs?"

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4 Scene Processing

To the uninitiated, the seizure of a clandestine lab may seem to be the culmination of the investigation. The long hours of surveillance, witness interviews, informant debriefings, information confirmation, and search warrant preparation lead to the excitement of the actual seizure. Yet, what seems to be the end of a process is, in reality, only the beginning. In some aspects, finding the clandestine lab is the easiest part of the investigation. Making sense out of what is found is far more difficult. The hazards indigenous to a clandestine lab crime scene make it impractical for even the best-prepared investigator to waltz through the scene, take a few photographs, throw the relevant evidence into a paper bag, and go back to the office.

The seizure of a clandestine lab should be a well-orchestrated event involving teamwork and timing. As in any team sport, each player has a specific job to do if the team is to be successful. The seizure of a clandestine lab requires that same teamwork and coordination. A clandestine lab seizure is a scheduled event requiring a number of small teams to perform their specialties in a coordinated sequence. These teams can be grouped into support teams and seizure teams.

Support teams are mostly comprised of non-law-enforcement personnel who provide services that may be needed during the various portions of the seizure process. These support teams include members from fire departments, emergency medical services, hazardous waste disposal companies, local health and environmental protection agencies, child protective services, and animal control. The circumstances surrounding the seizure can be used to predict exactly how many of these services may be needed.

Seizure teams are comprised of various law enforcement components with members that possess specialized training concerning clandestine labs. Each group has a specialized function. These groups are similar to those used in processing any major crime scene. Additionally, the hazards involved with clandestine labs require personnel and safety procedures to be incorporated

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into the scene processing procedures. Both explosive ordinance disposal (EOD) technicians and a forensic chemist who specializes in the manufacturing of controlled substances should be included in this group, even though they are not part of a typical crime scene response team.

4.1 Training

The hazardous nature of seizing a clandestine lab requires that people with specialized training be involved. The OSHA is in charge of enforcing the regulations established by Sections 1910.120, 1910.134 (respiratory protection), and 1910.1200 (hazard communication) of Chapter 29 of the Code of Federal Regulations (29 CFR …). Under 29 CFR 1910.120, an employer is responsible for providing training sufficient to educate employees concerning the dangers involved in working in a hazardous environment. As a result of such training, employees should be able to understand the hazards and risks associated with clandestine labs, understand the potential outcomes associated with an emergency response resulting from a clandestine lab, recognize and identify hazardous substances, and understand the need for additional resources when seizing a clandestine lab operation. The employer is also responsible for providing periodic (annual) refresher training to keep employees abreast of current information concerning hazards in their work environment.

To address these requirements, in 1986, the DEA began a training program designed to inform the personnel who investigate and respond to clandestine labs about the hazards involved in scene investigations. The objective of the program was to bring the DEA into compliance with 29 CFR 1910.120, 29 CFR 1910.134, and 29 CFR 1910.1200. The program initially covered DEA special agents, forensic chemists, diversion personnel, and task force personnel responsible for clandestine lab enforcement. The program included an initial 40-hour safety school, annual 8-hour recertification courses, a 32 h advanced course for site safety officers, and a comprehensive medical surveillance program.

There are a variety of training resources available to agencies that require safety training concerning clandestine labs. The DEA offered the initial 40-hour training course to law enforcement agencies that respond to clandestine lab scenes. Groups like the Clandestine Laboratory Investigators Association (CLIA) and the Clandestine Laboratory Investigating Chemists Association (CLIC) sponsored 8-hour recertification courses. Private companies, such as Network Environmental Systems, Inc. of Folsum, CA, were contracted to provide similar safety training to interested law enforcement agencies. State and local environmental quality agencies and fire departments can provide

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training on the handling of hazardous materials. The training information and resources an agency needs to educate its employees and comply with 29 CRF 1910.120 are available, if the agency is willing to explore the options that are available outside the traditional law enforcement training network.

4.2 Seizure Stages

Seizing and processing a clandestine lab are orchestrated events that involve arresting potentially violent individuals, then processing a crime scene and dealing with an environment that often contains hazardous chemicals. The involvement of hazardous chemicals in the crime scene necessitates that the seizure process be broken into a particular sequence of steps, with each step requiring a specific expertise. The steps of the seizure process include the preraid planning, the briefing, the entry and arrest, the hazard evaluation and abatement, the search and site control, and finally, the disposal of the hazardous waste.

4.2.1 Preraid Planning

Planning of a clandestine lab seizure starts long before the affidavit for a search warrant is written. Hopefully, the responsible agency identified the resources it will need to process the scene and established policies and procedures that delineate the notification mechanism necessary to coordinate their use during the seizure. Without proper planning, a clandestine lab seizure can turn into a nightmarish multiheaded dragon that law enforcement is not prepared to slay.

Development of an action plan and the policies and procedures required to safely process a clandestine lab is a labor-intensive process. It is also expensive to provide and maintain the training and equipment required. This can be a major administrative hurdle that an agency must take into careful consideration before jumping into the clandestine lab seizure pool.

To address this situation, many jurisdictions rely on a larger agency (i.e., a state or large metropolitan law enforcement agency) or pool their resources with other agencies to create a task force that specializes in investigation and seizure of clandestine labs. These alternatives provide the manpower and resources necessary to safely process clandestine lab scenes. Other agencies simply provide a response team that will process the scene. In many cases, the paperwork and physical evidence are handed back to the investigating agency for prosecution once the seizure process is completed.

There are a number of resources that should be identified as part of a clandestine lab response policy. Other law enforcement agencies in the region that may have beneficial investigative expertise should be sought out, such

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as the DEA or state narcotics enforcement agencies. Local fire department and emergency medical service (EMS) should be on standby in case of explosion, fire, or other medical emergency. Hazardous waste disposal companies need to be notified, and arrangements for payment must be initiated. Local health and environmental quality departments must be notified for public health reasons. Child protective services may be required, because children are frequently (albeit innocently) involved.

During the preraid planning stage, the primary investigator consults experts to ensure the known information is consistent with a clandestine lab operation. The investigator identifies the location of the suspected operation, provides information concerning why he believes there is a clandestine lab at that location, solicits the expert's opinion, and prepares an affidavit. The affidavit is then presented to a judge, who determines if there is sufficient evidence to establish the probable cause necessary to grant a search warrant.

Also described in the affidavit should be the hazardous nature of the chemicals involved and a formal request that they be disposed of professionally after being properly documented and after taking necessary samples. This statement notifies the court that there is a potentially hazardous situation, and that the law enforcement agency does not have the facilities to safely store or dispose of the chemicals seized. Further, the agency promises to make every effort to document and identify the items necessary to prove the State's case, while protecting the rights of the accused and maintaining public health and safety. This is quite a responsibility. It is imperative, therefore, that the document be carefully worded, because it must be all-inclusive.

It should be stressed that the bulk of the substance discovered will be *disposed of* rather than simply destroyed. "Destroy" implies intent to deceive. "Dispose" indicates a plan to mitigate hazards after the appropriate documentation and preservation steps have been taken. Even if the scene was properly documented and the necessary samples were taken, without the appropriate court authorization, the investigator may inadvertently cross the destroy/dispose line, negating all the evidence collected at the scene.

4.2.2 Briefing

All of the teams associated with the seizure of a clandestine lab should be brought together at the briefing. These teams include the entry team; the search team; EOD, and hazardous materials personnel; forensic chemists; fire department and EMS representatives; and representatives from other law enforcement agencies who may be assisting in the seizure. Personnel from local health or environmental quality departments and child protective services may be notified that their services may be needed, but their presence at the briefing is not required unless preliminary information exists indicating that they will be needed.

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During the briefing stage, the lead investigator provides a history of the case, identifying the suspects and the location of the operation. Clandestine lab experts and chemists brief the group on the hazards associated with the type of operation that they can expect to encounter. The staging area and command post locations are disclosed. A separate entry team briefing is performed shortly after the main briefing. Although this is a short stage of the seizure process, it is necessary in order coordinate the resources that will be needed to process the scene.

4.2.3 Entry and Arrest

The entry and arrest stage is definitely an "adrenaline-pumping" and exciting phase of the seizure process. It is also the most dangerous. The danger is in the team's lack of control over the unknown scenario. An entry team trained in clandestine lab seizures should be able to enter and secure a location in less than 2 minutes. However, because of the unpredictability of the situation, during those 2 minutes, anything can go wrong. The operator's mental state and level of drug-induced psychosis is a total unknown. The exact state of the manufacturing process is in question at the time of entry. There may also be booby traps present in any part of the lab or its environs.

The entry team has two functions: (1) secure all the personnel within the immediate lab area and (2) be the eyes and ears of the evaluation and abatement team.

The entry team's primary function is to secure the lab location. This is done by securing, detaining, and then removing any people from the lab area. Who these people are and their relationship to the lab are irrelevant. The seizure is not the time and the lab area is not the place to determine who is involved in the operation and who is at the location for some other reason, however unrelated. This initial step is mandatory for the people's safety and the safety of the personnel who will subsequently be processing the lab scene for physical evidence. Leaving detained personnel in the lab area is not wise, because it needlessly exposes everyone to the hazardous materials in the lab. Leaving desperate suspects in the lab area may further provide them with opportunity to attempt to destroy evidence, thus dramatically increasing the potential for a hazardous materials exposure.

The second function of the entry team is to act as the eyes and ears of the evaluation and abatement team. What the entry team sees, hears, and in some cases smells or tastes, is vital information used by the evaluation team to develop the scene's abatement plan. Members of an entry team trained in clandestine manufacturing techniques recognize the sights and odors of the chemicals and equipment commonly used in clandestine labs. The entry team is in the lab area such a short amount of time that their training must provide them with the ability to recognize significant items and to know the impor-

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tance of relaying said information to the abatement team for use in formulating the abatement plan.

Members of the entry team should be dressed in such a manner as to provide them with protection from the hazards they may encounter. In selecting the clothing worn during the entry phase, the team needs to consider inhalation and dermal exposures. All entry team equipment provides some level of dermal protection. However, there is a difference in philosophy when it comes to the use of respiratory protection. One extreme advocates not using any respiratory protection during the entry and arrest phase, reasoning that physical mobility, increased peripheral vision, and ability to give verbal commands outweigh the hazards of short-term exposure the team will experience. The other extreme counters that the lab atmosphere may contain lethal concentrations of any of many substances. Training and practicing entries while using the protective equipment helps overcome restrictions of the PPE. Wearing the equipment also adds additional psychological shock value to a dynamic entry as well to any people in the lab at the time, possibly buying critical time for the raiders. Anything that can be done to discourage last-minute tampering at the clandestine lab is recommended.

There are times at which a clandestine lab is encountered by law enforcement, fire departments, or EMS personnel during activities unrelated to clandestine lab investigations. The response to these situations is similar to that of an investigation-initiated seizure. An emergency on-the-scene briefing takes place that requires all professional expertise accumulated from other raids. The initial responders act as the entry team and remove all personnel from the area. They report to the site safety officer what they know about the inside of the lab area. In essence, they act as the entry team, becoming eyes and ears for the abatement team that they will then call.

4.2.4 Hazard Evaluation and Abatement

In clandestine lab investigations, the concept of crime scene processing changes once the site has been secured by the entry team. The main goal of the operation from this point is to create and maintain a safe work environment. If this goal is not achieved, the other goals of the operation may not be attained.

The site safety officer is responsible for implementation and maintenance of safe work practices at the scene of a clandestine lab seizure. His tasks include determining the hazard potential of the lab scene, establishing work zones, and determining the appropriate levels of protection for the various stages of the balance of the seizure operation. The total dynamics of the responsibilities of the site safety officer is beyond the scope of this book. However, in this section, some of the site safety officer's basic responsibilities

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will be presented so as to provide an understanding of what safety measures should be put into place.

The entry team's observations provide useful information that the site safety officer can use to develop the site control plan. Having this firsthand information from an objective source allows the site safety officer to make decisions in determining what PPE will be used, in establishing work zones, in ensuring safe work practices are being followed, and further, in making other vital decisions concerning workplace safety.

Interviewing the operator or other personnel who were removed from the lab area by the entry team (after arresting and reading them their Miranda rights if applicable), can supply useful information concerning what hazards may be inside. Some operators immediately invoke their right to remain silent and will not speak to law enforcement personnel. Other operators are perversely proud of their operation and are willing to tell someone who "admires the sophistication" of the operation all the details of what he is manufacturing and how he is doing it. The interviewer and the site safety officer must always consider the source of this information and bear in mind that the operator may have an interest in providing misinformation. His understanding of the technical aspects of the operation may be limited, and he may not know the real names of the chemicals involved. For these reasons, an expert in clandestine manufacturing techniques should sit in on the interview, if possible. The expert should not only know the technical aspects of manufacturing a wide variety of controlled substances, but also, more importantly, should recognize the slang terms for the equipment and chemicals that are commonly used in the various manufacturing processes. The lab operator may only know his chemical components by these names.

4.2.4.1 Site Control

Site control is a primary responsibility of the site safety officer. He determines what tasks will be performed and where, based upon an area's level of contamination. He also limits access to highly contaminated areas to personnel who have the required training and are wearing the appropriate PPE. The site safety officer will further divide the scene into areas based on level of contamination: hot, warm, and cold zones.

The hot zone consists of the area immediately surrounding the manufacturing operation or areas with open chemical containers. This area may only encompass the area surrounding an ice chest containing closed chemical containers, or it may incorporate an entire house, in which every room was used for some portion of the manufacturing operation. Who has access to the hot zone and what level of protection they should be wearing are the important issues. The hazardous nature of the hot zone mandates that access

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be limited and time inside be minimized. Once the hazards have been abated or removed, the hot zone can be downgraded to warm.

The warm zone is the area immediately adjacent to the hot zone. Its access should also be limited. However, the level of protection required here might not be as great. Items from the hot zone are moved into the warm zone, where they can be processed under controlled conditions. Hot zone workers take rest breaks and are decontaminated in the warm zone. Emergency rescue personnel stage here, in case an accident requiring a rescue occurs in the hot zone. Even though the hazards are not as great, access to the warm zone should be limited to trained personnel wearing appropriate PPE. The warm zone can be downgraded to a cold zone once the sampling process is completed, and the chemical containers have been sealed and segregated.

The cold zone is a hazard-free zone where the command post is located. Here, the lead investigator and site safety officer coordinate the seizure activities. Any eating, drinking, and smoking should only take place in the cold zone to reduce the possibility of ingesting hazardous materials. An access point to the warm and hot zone is established here to ensure that only authorized personnel enter the contained area and to document who had access to the crime scene for later court purposes.

4.2.4.2 Personal Protective Equipment

In conjunction with site control, the site safety officer must establish the levels of personal protective equipment (PPE) that will be required during the various stages of the processing operation. He relies on information from the entry team to determine the level of protection the evaluation team will require during the evaluation and abatement phase. The evaluation team's information will be used to determine protection levels required for subsequent stages.

PPE levels range from "A" through "D." Level A provides the greatest level of protection for all entry routes. Level D protection is not much more than work clothes.

Level A PPE is total encapsulation, and it provides the highest level of respiratory and skin protection. It is used in atmospheres that are IDLH and require extreme dermal protection from compounds to which skin exposure in small concentrations will result in impairing or life-threatening health effects. The use of supplied air provides for operation in oxygen-deficient environments.

Level B PPE provides protection similar to level A, but the configuration of the equipment does not offer quite the level of dermal protection. However, the respiratory system is totally protected. This level of protection is recommended for the assessment phase of operational clandestine labs. Some agencies utilize this level of protection for their entry team.

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Level C PPE provides Level B barriers to dermal exposure, but respiratory protection has been downgraded from supplied air to an air purification respirator. This level of protection is appropriate when the composition and concentration of the work atmosphere is known and continually monitored. Level C protection is not appropriate for oxygen-deficient atmospheres. However, it is appropriate for work in ventilated areas in which the chemical composition of the hazardous materials involved is known.

Level D protection is used when the potential for chemical contact is minimal. It can be equated to industrial work clothes and is appropriate to protect the worker from incidental exposures.

The evaluation and abatement team has two primary functions: (1) identify and neutralize any potential hazard within the hot zone and (2) create a less hazardous environment so further scene processing can occur.

Using the appropriate PPE, the evaluation and abatement team will enter the hot zone to identify and evaluate the potential hazards. Team personnel should have a solid knowledge of improvised explosive devices (IEDs; i.e., booby traps), clandestine manufacturing techniques, and hazardous materials chemistry. A forensic chemist and an EOD technician form a complementary team that can evaluate and abate the identified hazards. At minimum, they should have equipment that can monitor the atmosphere's oxygen content and the level of flammable and explosive vapors. They may also use equipment that can identify and quantify levels of a variety of specific hazardous materials. The site safety officer is ultimately responsible for selecting the equipment used to evaluate and monitor the hot zone's atmosphere.

Abatement can begin once the hazards have been identified. The EOD technician performs "render safe" operations to items with explosive potential. A forensic chemist trained in clandestine manufacturing techniques can shut down active chemical reactions (see Table 4.1). Open chemical containers are sealed, and confined spaces are safely ventilated. Once these abatement procedures have been conducted and a safe work environment has been established, the actual processing of the scene can begin.

4.2.5 Scene Processing

The processing of a clandestine lab scene is a unique combination of processing a crime scene and a hazardous materials incident. Therefore, two considerations must be balanced during this phase. First, the integrity of the evidence must be maintained. Second, exposure to the hazardous materials must be kept to a minimum. These tasks can be accomplished simultaneously by implementing three steps: planning, documenting, and sampling.

The number of people used to process the lab area should be kept to a minimum to accomplish both of these tasks. Minimizing the number of people in the hot zone minimizes the number of people exposed to the highest

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Table 4.1 Reaction Shutdown Guide

- Determine whether the reaction is being heated, cooled, or both.
- Remove heat from the reaction vessel.
- Maintain cooling to the reaction until the reaction appears to have gone to completion and the vessel is cool to the touch.
- Remove obstructions and ventilation tubing from the top of condensing columns.
  - Remember that cooling reactions can create a vacuum that could hamper the dismantling of the apparatus.
  - Note that if the reaction is being vented into water, the vacuum created by the cooling reaction could draw the water into the hot reaction mixture, leading to a violent reaction.
- Turn off at the source, if necessary, compressed gas containers connected to a reaction vessel.
- Allow the pressure of the reaction vessel to naturally reduce to atmospheric pressure.
  - Remember that release of pressurized contents of the reaction vessel may create a toxic exposure.
- Bring systems under vacuum slowly back to atmospheric pressure.
  - Note that water or oxygen in the air may react violently with the chemicals in vessel under vacuum.
- Allow filtration processes to naturally go to completion.

contamination levels. Because the lab area is generally a confined space, increasing the number of people in the area increases the potential for an accident. A small processing team can more efficiently perform the sequence of tasks required to preserve the integrity of the evidence.

4.2.5.1 Planning

Planning is an essential step of any process. The first step of establishing the plan for processing the clandestine lab scene is to touch nothing, instead using the powers of observation. Every clandestine lab is different, and the person in charge of the scene processing must avoid the desire to start moving things from the hot zone before evaluating the totality of the scene. He should walk through the scene, making mental notes and asking himself questions like: What type of lab is this? What process(es) is apparent? Which chemicals are present? What equipment is present? What does the operator seem to be trying to make? Once a picture of what the operator was trying to make and how he was trying to make it has been developed, a plan of how to process the scene to prove an hypothesis can be developed. Practical examples in Chapter 9 demonstrate why a walk-through should be conducted before processing every lab or making assumptions.

A word of caution is needed at this point. The objective of a forensic investigation is to allow the physical evidence to dictate the facts of the case. The scene should be looked at in a totally objective light. The physical evidence at the scene needs to drive how the search is conducted. The scene

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processing team needs to guard against getting caught up in the "get the bad guys" mentality of the seizure and focus on identifying, collecting, and preserving the physical evidence that indicates the presence of a clandestine manufacturing operation. This is not to say the team cannot focus on collecting evidence characteristic of a clandestine lab that also has common uses. However, investigators should also be careful not to place an illicit meaning on an innocuous item if the scene does not justify it.

4.2.5.2 Documentation

Documentation is essential to preserve the integrity of the evidence, because most of the evidence will be disposed of fairly quickly because of its hazardous nature. To give a complete picture of the clandestine operation, a combination of techniques is necessary. One augments and complements another, thus providing a thorough record of the existence of the evidence, even though it is no longer available. Methods of documenting a clandestine lab scene include photography, videotape, scene interviews, field notes, sketches, and inventories. Each has its strengths and weaknesses.

The documentation of the scene should take place throughout the seizure process. It can be initiated during the evaluation and abatement phase but usually begins during the initial walk-through, when photographs and field notes are taken. A sketch of the scene is made to supplement the photographs. This is followed by itemizing and taking inventory of the specific items that are removed from the scene. A final documentation of how the seized items are disposed of and the remaining items are stored is done.

4.2.5.2.1 **Photography.** Photography is the traditional method of recording and documenting crime scenes. It captures the way the scene first looked and documents specific items and situations. The initial series of photographs should tell a story and walk the viewer through the scene and portray the scene as the photographer observed it.

Photo documentation can commence once the scene has been secured, the hazards abated, and the atmosphere deemed safe. General overall photographs should be taken to depict the lab area as it was originally encountered. These photographs should be taken from multiple angles before anything is moved. If possible, a series of photographs creating a panoramic from a fixed point should be taken. This creates an overall view of the lab as the photographer encountered it. This should be followed by taking close-ups of specific items in their original location. Reaction apparatuses should be photographed before they are dismantled.

A photograph should be taken of every item or group of items seized. This can be done in the warm zone under controlled conditions. These photographs serve as corroboration to the written field notes and official

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inventory. They should be as clear and accurate as possible, because they are the official record. Photographs should be taken of the samples with their original container to demonstrate where the sample item was removed.

Individual item photographs should contain identifying information. This information should minimally include the case number, the exhibit or item number, and the date. A ruler scale should be included if volume calculations will be made using the geometry of the container at a later time.

A photo log should be maintained. This documents who took the photographs, when they were taken, what the photographs depict, and why the photo was taken. The investigator must remember that the photographs are more than pictures used to enhance his memory at some point in the future. They are evidence, and as such, they have the same requirements concerning maintaining the chain of custody, as does any other physical evidence seized from the scene. Photographs are discoverable evidence and must be treated as such. To complete a photography packet, the back of each photo should be labeled with the case number, photographer's ID, the photo number, and the total number of photos taken at the scene. This information should correspond to the information on the photo log.

The introduction of digital photography into clandestine lab processing has provided the investigator with tighter control over the images he generates during his scene investigation. It is suggested that an investigator using digital photography copy the images from his camera directly to a "read only" file format on a removable storage format (compact or floppy disc) to serve as the permanent record, much the same way negatives serve as the permanent record for traditional film photography.

4.2.5.2.2 Videotape. Videotaping the scene has become a popular method of visually documenting clandestine lab scenes. It is a real-time way to demonstrate how the site was originally found and it provides the viewer a sense of actually being at the location, as the photographer pans through the scene.

The audio component of videotape can be a help or a hindrance. If the person providing the commentary is knowledgeable about what is being viewed, it can augment the visual presentation. However, as exhibited in the Practical Examples in Chapter 9, the audio support did not accurately describe what was being viewed. This conflict caused problems during the expert's trial testimony. The videographer should, therefore, refrain from commenting if he is not sure what he is looking at.

As with a traditional photograph, videotape records of the scene are discoverable evidence. Every effort needs to be made to preserve the unaltered tape and maintain its chain of custody. Copies can be made as needed. However, the original tape must be fixed so that it cannot be altered or erased. It should also be labeled with the pertinent case information.

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4.2.5.2.3 Field Notes. Field notes are taken on or about the time the clandestine lab is processed. They are another form of documentation that is used to augment the others. Field notes are used to supplement the visual images of the photographs or videotape. Field notes can be written at the scene, dictated into a recorder, and transcribed at a later time, or they can be written at the office immediately after the fact.

Field notes can take a variety of forms and are used to address the questions of who, what, when, where, why, and how concerning the crime scene investigation. Who assisted in processing the scene and had access to the evidence? What items were seized? When was the lab entered or seized? Where were the seized items located? Finally, why were certain items seized and not others?

The three basic forms of field notes are worksheets, narrative descriptions, and sketches. Each has its strengths and weaknesses. Most clandestine lab investigators utilize some combination of the three.

All forms of field notes must be treated as part of the written record and are subject to discovery. Each page should have the same basic information as a photograph and identify the author (name, initials, or ID number), case number, date, page number, and total number of pages.

Worksheets are unique to a specific task. They provide a convenient method of documentation and a format to walk a person (and later a jury) through the various phases of a clandestine lab seizure. There are worksheets or checklists for preraid planning and the hazard evaluation and abatement phases. Some investigators designed worksheets to assist in documenting specific items seized from the lab. Worksheets are good for noting routine responses to required repetitive tasks or confirming seizure of the same evidentiary items. However, worksheets can be ineffective if their parameters do not address the scene scenario. Worksheets with a narrative section can be used to provide additional information in situations that may be outside the scope of the worksheet.

A photo log form is an example of a worksheet used to supplement the photographs taken at the scene. This form contains fill-in-the-blank and narrative sections. There can be sections for time, date, photographer ID, photo number, and roll number. The section that describes the photograph and its significance is a short narrative.

There is no right or wrong way to take narrative notes. Notes can be as short or as long as the author desires. They can be short phrases used to assist the investigator's recall at a later time, or they can be multisentence explanations of how the author perceived the operation functioned. As long as the author can place his thoughts on paper in such a way that he can decipher them at a later time, the goal has been achieved. The only rules regarding narrative notes are that they be preserved and that each page contains appropriate case information.

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Photographs and videotape document the actual presence of items at the scene. However, because they are two dimensional, they poorly demonstrate where things were located in relationship to other items. Wide-angle lenses or panoramic film might provide the answer, but their costs are rarely within the budgets of most law enforcement departments. Sketches, therefore, provide a means of demonstrating a spatial relationship that photographs do not. By combining these two-dimensional formats, a three dimensional perception that accurately depicts the scene can be developed.

Sketches can be as detailed as the drafter deems necessary. They can be used to provide a basic special relationship between items at the scene using visual approximations to determine distances. Sketches can also be extremely detailed, using exact measurements from fixed points to create a scaled drawing of the scene. As with all forms of field notes, information concerning the case and the drafter's ID should accompany the sketches.

4.2.5.3 The Search

Hot zone searches are handled differently from the balance of the scene because of the presence of hazardous materials and possible contamination. People who have not received specific clandestine lab training can process searches of warm and cold zones adjacent to the lab area using standard crime scene search techniques. However, a clandestine lab expert should be available to provide technical advice when clandestine-lab-related items are found in these areas.

Once the original condition of the scene is documented, the search and inventory can commence. The search is necessary to locate and identify the items of physical evidence necessary to establish the clandestine manufacture of controlled substances. Experts in a variety of clandestine manufacturing techniques should conduct the search. These experts are able to recognize the evidentiary significance of ordinary items at the location. The searchers should also have been provided the safety training required by 29 CFR 1910.120. Again, the hot zone is a hazardous materials scene that may also be a confined space, so the number of people conducting the search should be kept to a minimum.

There are three approaches concerning the search of the lab scene. First is simply utilizing a methodical approach. Second is using a "clear the area" approach. Last is implementing the "sample in place" approach. Personnel safety and legal considerations are used in selecting a search method.

First and foremost, a clandestine lab is a crime scene. Every effort should be made to preserve the integrity of the physical evidence. To this end, an organized search of the lab area is needed. A methodical search allows the processing team to sequentially move through the scene and identify, document, collect, and preserve each evidentiary item located. Once the item's

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location is noted, the time it is moved from the hot zone to the warm zone for further processing is documented. This approach can be time consuming and, thus, increases the length of time the search team is in the dangerous hot zone.

The "clear the room" approach minimizes the amount of time the search team is in the contaminated environment. This approach moves all of the items from the hot zone to the warm zone for processing, without detailing the exact location and time each item is removed from the scene. The overall photos and the sketch document the scene's original condition. Supplemental photos during the inventory and sampling in the warm zone serve to document individual items.

The third method is a compromise of sorts. The "sample and go" approach reduces the search team's contact with the hazardous materials by not moving them from the hot zone to the warm zone. The scene is searched, inventoried, and sampled without significantly moving any items. Once the team has completed its search and documentation of the hot zone, it is turned over to the waste disposal company. Using this method can increase the time in the hot zone but minimize the situations in which accidents while handling hazardous materials can occur.

The search of uncontaminated areas adjacent to the lab area can be conducted using traditional crime scene search techniques. However, a forensic chemist and EOD personnel should be readily available. It is common for searchers to find chemical or explosive devices in an area surrounding the location. Hazardous materials have been located in children's bedrooms, bathrooms, closets, attics, outbuildings, and vehicles parked on the street. The specialists are needed to evaluate the evidentiary significance of the items, to take the actions necessary to address any hazards, and also to preserve the evidence.

4.2.5.3.1 Inventory. The need for an accurate inventory of the chemicals and equipment seized from a clandestine lab goes beyond establishing the elements of the crime. The inventory is used to establish facts and render opinions concerning the lab that are not apparent at the scene. Evaluating the inventories of the seized items can identify manufacturing methods used and create estimates of production (see Chapter 6).

All law enforcement agencies have some type of form (worksheet) for documenting the items seized during the execution of a criminal search warrant. Depending on the agency's policies and procedures, these forms may or may not adequately depict the type and amount of chemicals and equipment seized. Therefore, it is suggested that a separate detailed inventory be taken to accurately document the types and amounts of chemicals and equipment seized, so that experts can objectively evaluate the information

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away from the hectic environment of the seizure and render opinions concerning the overall manufacturing operation.

4.2.5.3.2 Sampling. Even small operations can create a significant number of evidentiary items. The hazardous nature of the chemicals and contaminated equipment requires special storage conditions. Most law enforcement agency's property rooms are not equipped to handle the volume of evidence that can be generated by a clandestine lab. They also do not have the ventilation and safety equipment necessary to safely store and dispose of such hazardous waste seized. Therefore, taking evidentiary samples from the scene of a clandestine lab is a necessity. The balance of the items can be properly disposed of once properly documented.

It is highly recommended that a forensic chemist who specializes in clandestine labs perform the sampling. His knowledge of the various clandestine manufacturing methods allows him to select the significant items, while at the same time, keeping the samples to a manageable number for the forensic laboratory. He can recognize subtle differences in unknowns and select samples that will accurately depict the seized operation. He also has the training to allow him to safely handle the hazardous chemicals that require sampling and to recognize the materials that should be handled with kid gloves, or those that should not be handled.

Local statutes and case law will determine what to sample and how much to take. The guidelines assist in indicating which items should be sampled. Suspected controlled substances should be seized in total and submitted for examination, while explosives should be sampled. All suspected precursor chemicals should be sampled; their identity assists in determining the synthesis route used and in estimating the amount of final product that could be produced. Taking samples of reagents and solvents should be left to the discretion of the on-scene chemist. The identity of reagents assists in determining the synthesis route, and the identity of solvents can assist in determining the extraction method used.

The fate of labeled containers is not as well defined as one would think. It is a good assumption that the contents of a factory-sealed commercial container are what the label reports them to be. Opened containers with commercial labels may or may not contain what is on the label, and the on-scene chemist should treat them accordingly.

The only difference between an unlabeled container and a container that the operator has labeled is that the label reflects only what the operator originally placed into the container; the contents of an operator-labeled container may have been changed any number of times since the label was originally applied. Therefore, the container should be treated as an unknown.

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The following is a general guide for sampling chemical containers. It is unnecessary to sample factory-sealed commercially labeled containers if they have been properly documented. The sampling of open containers with commercial labels is up to the discretion of the on-scene chemist. Commercially labeled containers with contents that are not consistent with the label should be sampled. Samples should be taken from all containers with altered commercial labels, handwritten labels, and without labels; and identification of such labels should always be indicated.

Reaction mixtures, extraction mixtures, waste liquids, and sludge contain a wealth of information in a single location. Lack of a label of any type indicates nothing. These mixtures may contain many, if not all, of the precursors, reagents, final products, and by-products for a given synthesis route. This wealth of information from a single source necessitates the sampling of these mixtures. Nothing should be ignored.

Glassware and equipment found at a clandestine lab scene may or may not contain residues that will provide information concerning what was being produced or the synthesis method being used. For safety reasons, the entire piece or a number of pieces of glassware or equipment must be submitted to the forensic laboratory if the identity of residue is deemed critical. This will provide a controlled environment for sampling and examination.

The search and inventory method used will determine where the samples will be taken. The "sampling in place" method is just that: samples are taken at the items' original location. Other methods move the items to an evidence-processing location, where the conditions of the sampling can be more controlled. Trained personnel wearing the appropriate PPE should do any sampling in a well-ventilated area.

The hazardous nature of the samples requires special packaging. Packaging should take into account the chemical properties of the samples and be designed to minimize the hazardous exposures and cross-contamination if a sample container is broken. Finally, the packaging should provide information concerning the sample's identity. This information should include the case number, item number, date, and any relevant information concerning the sample, such as its pH, field test information, original volume, or weight. Even the item's proximity to another item may be important. In Table 4.2, information concerning the appropriate packaging of clandestine lab samples is provided. Guidelines for items to include in a sampling kit are provided in Table 4.3.

The question of how many samples are required is invariably asked. As with every other aspect of clandestine lab investigation, there are a number of ways to approach the situation. All represent merely a difference in philosophy. The sampling camps can be divided into their extremes: one camp advocates taking only the minimum number of samples necessary to establish

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Table 4.2 Sampling Guide

General Considerations

- Consult local statutes and case law concerning the type and amount of samples required.
- Conduct sampling in a well-ventilated area, preferably away from the lab area.
- Photograph all samples with the original container.

What to Sample

- Seize the entire amount of controlled substances.
- Sample all reaction mixtures.
- Take random samples of waste material.
- Sample unlabeled chemical containers.
- Leave the sampling of reagents and solvents to the discretion of the on-scene chemist.
- Sample each phase of a multiphase liquid.
- Leave the sampling of commercially labeled containers to the discretion of the on-scene chemist.
  - Assume that factory-sealed containers contain what the label reports.
  - Note that open commercially labeled containers may or may not contain what the label reports.
- Leave the sampling of glassware to the discretion of the on-scene chemist.

Sample Packaging

- Package liquid samples in glass vials with acid-resistant screw caps and place inside a sealed zip-lock plastic bag.
- Place solid samples into a sealed zip-lock plastic bag.
- Place solid and liquid samples inside a second sealed zip-lock plastic bag.
- Mark the outer-sealed zip-lock plastic bag with the appropriate case information.
- Place individual items into a single container filled with an absorbent material for transportation and storage.

Table 4.3 Sampling Kit

Necessary Items	Desirable Items
Camera (35 mm or digital)	Chalk or dry-erase board with markers
30, 1–2 oz glass vials with acid-resistant screw caps	Scales (1 kg and 100 kg capacity)
30, 25 ml disposable volumetric pipettes	Field test kit
60, 4" × 6" zip-lock plastic bags	• Disposable culture tubes
3 pipette bulbs	• Disposable plastic eye droppers
100, 3" × 5" index cards	• Wooden spatulas or applicator sticks
Marking pens	• pH paper
Tape	• Premixed reagents (See Table 4.4)
Ruler or tape measure	
Worksheets or notepads	

the elements of the crime, and the other advocates sampling everything in sight. What actually occurs is discretionary sampling by the on-scene chemist based on his experience and the law enforcement department's expectations based on similar venues.

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Taking a minimum number of samples streamlines the scene-processing phase. Minimizing exposure to toxic materials, it potentially reduces the analysis time required by the forensic laboratory. The downside to this minimization is that if proper or sufficient samples are not taken at the scene, there is no backup physical evidence to examine. Additional samples required to supply information that would provide a different perspective to the operation are, thereby, forever unavailable, because once law enforcement leaves the scene, a hazardous waste disposal company usually removes bulk items.

Another consideration of minimization is the possible appearance of deception. The argument could be presented that by not "completely" sampling the scene, the investigators were attempting to hide evidence that would exonerate the suspect or would be intentionally misleading as to what was actually occurring at the location. Are these valid arguments? They are probably not. Can they arise? Yes, they can and do.

The other extreme is to sample everything. If the total operation consists of three or four items, sampling everything is not unreasonable. However, sampling 30 unknown liquids with similar colors and chemical characteristics may be excessive, expensive, and time-consuming. Still, a guiding principle may be that it is better to have too many samples than not enough. The forensic laboratory may choose not to analyze a sample if the examiner feels that no additional information will be derived from the examination. However, it should always be remembered that the laboratory cannot analyze what it does not have.

4.2.5.3.3 Field Testing. Field testing is used to address two basic questions: What is it? How much is there? However, existence leads to follow-up questions of where and when is it appropriate to answer the first two questions?

The "how much is there" question needs to be addressed at the scene. In order to establish the amount of controlled substance that could be produced by the operation, the weights and volumes of the precursor and reagent chemicals need to be determined. The original volumes of reaction and extraction mixtures, combined with the results of laboratory analyses of the samples, can be used to calculate the amount of controlled substance that was in the container at the time of seizure.

The volume estimate of commercially labeled containers is relatively straightforward: it can be assumed to be what is reported on the label. Estimates can be made for commercially labeled containers with contents that appear to be consistent with the labeling information (e.g., 500 g jar approximately half full or 500 ml bottle approximately 25% full). By physically weighing the container and subtracting the weight of an empty container, or one of the same approximate size and type, more accurate estimates

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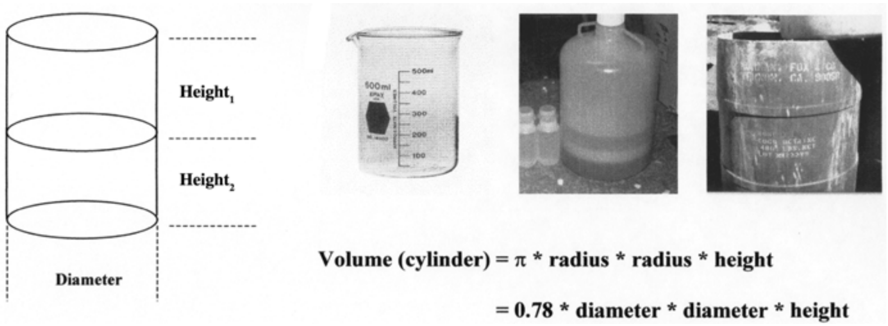

Figure 4.1 Cylinders.

of solid weights can be obtained. Measuring the dimensions of the liquid in the container and using basic geometry to calculate the volume of the liquid can lead to more accurate liquid volume estimates.

Determining the exact volumes of reaction and extraction mixtures and unknown liquids is more critical. These liquids potentially contain a controlled substance, the amount of which may be used in the sentence determination phase of a trial, if the suspect is convicted. Knowing the original volume of a container will enable the forensic chemist to calculate the amount of controlled substance that was in it.

All equipment and chemical containers found at clandestine lab scenes can be divided into one of three basic geometric shapes: cylinders, cones, or spheres. Cylinders (Figure 4.1) are the basic geometric shape of beakers, bottles, and drums. Erlenmeyer flasks, vacuum flasks, and separatory funnels are shaped like cones (Figure 4.2). Reaction flasks have a spherical shape (Figure 4.3). Knowing the basic shape of an object and the measurements of the liquids within it allows the actual volume to be calculated with preexisting mathematical formulas. Demonstrated in Practical Applications and Examples in Chapter 9 is how these calculations can be used in the field.

The accuracy of the answer to the "what" question is not as important to know at the scene. The exact identity of any given item cannot usually be established under field conditions. However, the ability to classify compounds and mixtures provides a means to efficiently group similar substances and streamline the sampling process. For example, knowing the basic chemical and physical properties of a liquid can tell the sampler whether the sample is a reaction mixture or a waste material. Knowing the relative density of an organic liquid will provide insight as to whether the final product will be on the top layer or the bottom. Certain chemical color tests can be used to provide presumptive information as to whether an item contains a controlled substance, or they can be used to indicate the item's reactive proper-

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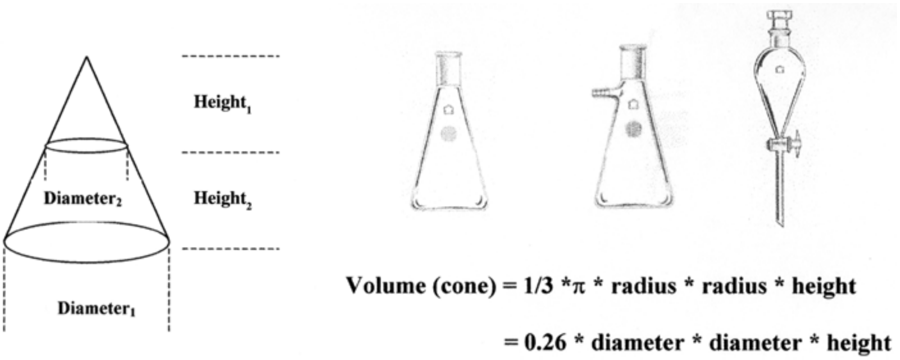

Figure 4.2 Cones.

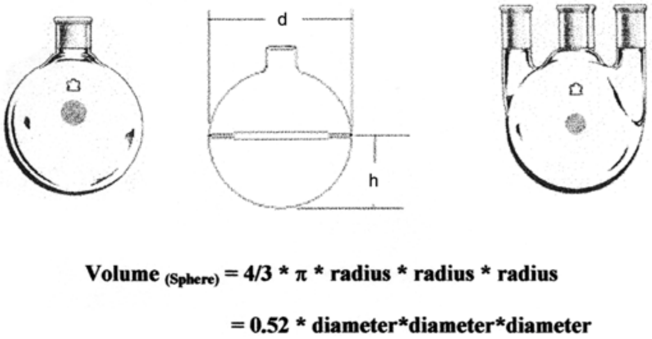

Figure 4.3 Spheres.

ties. For all of these determinations, a forensic chemist's experience on the scene is invaluable.

Described in Table 4.4 and Table 4.5 are the contents of field test reagents and some of the generic results that can be obtained from reactions with chemical color tests in the kit. As with sampling, field-testing should only be performed by trained personnel under controlled conditions. Trained personnel know the sequence of field tests for a particular sample that is necessary to provide an accurate picture of the samples' physical and chemical properties as well as potential contents. They are also aware of the subtle differences between a positive and a negative test result. Many field tests involve a sequence of chemical reactions, and the use of trained personnel reduces the likelihood of an incompatible chemical reaction.

4.2.5.4 Disposal

The final phase of a clandestine lab seizure must be considered long before the investigation begins. The following question must be asked and addressed:

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Table 4.4 Field Test Reagents

Reagent	Formulation
Marquis	0.5 ml formaldehyde combined with 30 ml of sulfuric acid — store in amber glass vial with acid-resistant cap
Cobalt thiocyanate	5% aqueous solution of cobalt thiocyanate (CoSCN)
Copper sulfate	• Solution A: 5% aqueous solution of copper sulfate ($CuSO_4$); Solution B: saturated aqueous solution of sodium bicarbonate ($NaHCO_3$)
Dinitrobenzene	• Solution A: 2% solution of *m*-dinitrobenzene, in reagent alcohol; Solution B: 5% solution of sodium hydroxide (NaOH)
pDMBA	• Solution A: 2% solution of *p*-dimethylaminobenzaldehyde (pDMBA); Solution B: concentrated hydrochloric acid (HCl)
Silver nitrate	5% aqueous solution of silver nitrate ($AgNO_3$)
Barium chloride	5% aqueous solution of barium chloride ($BaCl_2$)
Diphenylamine	• Solution A: 1% aqueous solution of diphenylamine; Solution B: concentrated sulfuric acid (H_2SO_4) — store in amber glass vial with acid-resistant cap
Thymol	• Solution A: 1% aqueous solution of thymol; Solution B: concentrated sulfuric acid (H_2SO_4) — store in amber glass vial with acid-resistant cap
Nesslers	• Dissolve 5 g of potassium iodide (KI) and 10 g of mercuric chloride in 50 ml of deionized water; dissolve 20 g of potassium hydroxide (KOH) in 50 ml of deionized water; combine solutions
Methanol/sodium hydroxide	1 N solution of sodium hydroxide (NaOH) in methanol

What is going to happen to the hazardous waste generated as a result of the seizure?

In the early days of clandestine lab investigations, waste disposal was not an issue. Chemicals were routinely poured down drains or on the ground, thrown into the trash, burned, blown up, or simply submitted to the police property room for indefinite storage. These actions were taken ignorantly, yet innocently, without regard to chemical compatibility, the health and welfare of the people who would be subsequently exposed, or the environmental impact.

Since then, numerous safety and environmental regulations have been established to protect the health and welfare of workers, the general population, and the environment. Law enforcement is not exempt from these regulations. In many cases, the lead agency legally becomes the "generator" of the hazardous waste produced as a result of a seizure. As the generator, they have a "cradle-to-grave" responsibility to ensure that the waste generated by the seizure is disposed of in such a way as to not adversely impact the

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Table 4.5 Field Test Reactions

Reagent	Color	Indication
Marquis	Orange	Phenethylamines, phenylacetic acid
	Purple	Opiates, MDA, MDMA
Cobalt thiocyanate	Blue	Cocaine HCl, PCP, meperidine, lidocaine
Copper sulfate	Blue	Ephedrine, pseudoephedrine, lidocaine
Dinitrobenzene	Purple	Phenyl-2-propanone (P2P)
pDMBA	Purple	LSD, indoles
Silver nitrate	White	Cl^-, CO_3^{-2}, SO_3^{-2}
	Crème	Br^-
	Yellow	I^-, PO_4^{-3}
	Brown	OH^-
	Black	S^-
Barium chloride	White	CO_3^{-2}, SO_3^{-2}, SO_4^{-2}, PO_4^{-3}
Diphenylamine	Blue	NO_3^-, ClO_3^-, ClO_4^-, nitro compounds, oxidizers
Thymol	Green	NO_3^-
	Brown	ClO_3^-
	Red	RDX, HMX
	Blue green	PETN
Nesslers	Orange	NH_4^+
Methanol/sodium hydroxide	Red to orange	TNT
	Blue to brown	DNT
Sulfuric acid	Yellow orange	ClO_4^-

environment or public health. In many cases, this means that the seizing agency takes on the financial burden of cleaning the chemical contamination that resulted from the clandestine operation.

This potential liability has sometimes made small agencies reconsider clandestine lab enforcement operations. The cleanup costs for the smallest operations can easily run into thousands of dollars. A medium-size operation could have devastating effects on the police department's budget. For this reason, task forces have been created and funds established to address the financial burden associated with clandestine lab seizures.

The generator is ultimately responsible for the waste that is generated from the lab. Therefore, it is imperative that the disposal company chosen be reputable in every way. When evaluating waste disposal companies, the low-bid approach taken by many government agencies may not be the optimum method of selection. The company selected should have the appropriate federal and local licenses to handle hazardous waste. They should be transporting it to approved facilities for proper disposal using approved methods. Finally, the personnel the waste disposal company sends to the site should have clean criminal histories, especially in the area of drug abuse.

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4.3 Summary

The seizure of a clandestine lab often goes beyond the scope of a traditional crime scene. The dynamics of hazardous materials involved must be taken into account, and the safety of the personnel processing the scene must be paramount. However, during the process, personnel cannot lose sight of the goal of preserving the physical evidence that indicates the existence of a clandestine laboratory. This balancing act can be accomplished through the use of specialized teams with specific functions. The number of people involved can give the process a circus-like atmosphere; however, with a documented set of policies and procedures delineating the responsibilities of each of the teams in place, order can be derived from what appears to be total chaos. The array of arbitrary unknown items can be sequentially identified, documented, preserved, and properly disposed of in such a way that the health and safety of all parties is protected, while at the same time, a forensic investigation can be conducted that will prove or disprove the existence of criminal activity.

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Laboratory Analysis 5

To analyze samples from clandestine labs, a variety of scientific techniques are employed. These techniques range from simple chemical color tests to the use of x-ray and infrared energy to elicit the compound's chemical fingerprint. The type of test used depends upon the information desired from the sample and the burden of proof required to establish its identity.

In this chapter, the techniques used to analyze evidentiary samples from clandestine labs are specifically addressed. A number of technical issues will be presented in a basic format to provide an understanding of the analytical process for readers. The purpose of this chapter is not to provide a detailed discussion concerning the theory of a particular examination technique. It is simply to present the options available to the analytical chemist. By reading this chapter, the investigator can gain an understanding of what examinations to request when submitting his evidence for examination. Also assisted will be the attorneys involved in the case, by providing them information concerning why certain tests were used as opposed to others.

The laboratory analyses of samples taken from the scene of a clandestine lab are the link between the investigation and the opinions. It provides the scientific proof that corroborates the investigator's theories and is used to justify the opinions rendered in reports, deposition, and testimony. Without complete and thorough laboratory analysis, the case may be unresolved.

The laboratory analysis of evidence is more involved than simply identifying a controlled substance. Identification of the components of the sample matrix may be just as important. A complete analysis is important in establishing the manufacturing method. It is not absolutely necessary. However, if the chemist's analysis is not complete, it may be implied that he is not qualified to perform the analysis or that he has something to hide. The lack of a complete analysis may also affect other aspects of the investigation or prosecution of which the chemist is not aware.

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It is not sufficient to say that the clandestine lab operator was using a particular method simply because some or all of the ingredients were found at the site. The presence or absence of a particular precursor or reagent chemical cannot be established beyond a reasonable doubt without laboratory examination. The relabeling or lack of labels on containers at the scene makes the identity of the chemicals at the location questionable.

The same holds true with reaction mixtures. The chemist should identify the ingredients within the reaction mixture. The fact that a chemical or chemical container was located at the scene does not establish its presence in a reaction mixture. It only provides the chemist information he can utilize in developing his analytical scheme.

5.1 The Chemist

The chemist performing the laboratory examinations should specialize in clandestine lab analysis. In bookkeeping, all CPAs are accountants, but not all accountants are CPAs. The same is true with forensic chemists. All clandestine lab chemists are forensic chemists, but not all forensic chemists are clandestine lab chemists. The clandestine lab chemist has additional training in clandestine manufacturing techniques as well as in inorganic analysis. This allows them to expand their analytical scheme to identify all the chemicals used in the manufacturing process. His analytical scheme is geared to identifying the manufacturing process, not just the controlled substance involved.

The chemist's role in a clandestine lab investigation requires a different thought process when approaching his analysis. He approaches each sample as if he has to tell to a jury what components are in the sample and how they fit into the manufacturing process. From an investigative standpoint, his analytical approach is geared toward profiling the sample to provide the investigators information concerning the sample's composition, so the investigators know what components to look for.

There are two schools of thought concerning which forensic clandestine lab chemist analyzes the samples once they enter the laboratory. One school has the chemist who processes the crime scene analyzing the samples, essentially, a "cradle-to-grave" approach. The other school has an independent chemist analyze the samples once they reach the laboratory. This school theorizes that it should not matter who does the analytical work, as long as the person is trained in clandestine lab analysis. Practical applications in Chapter 9 contain examples of actual situations. These applications demonstrate the ramifications that need to be considered when addressing how many chemists should be assigned to process a clandestine lab case.

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5.1.1 Single Chemist

Having a single chemist process the scene and subsequently analyze the samples can streamline the analytical process. The scene chemist understands the relationship between samples and the importance of each in the investigation. This broad understanding produces an intuitive prioritization of the samples based upon the direct knowledge of the sample's origin. If a sample's analytical results are consistent with the chemist's on-scene theories, analysis of similar subsequent samples may not be necessary. If they are not theories, analytical schemes and opinions may need to be modified to follow the direction in which the evidence leads.

The scene information is extremely useful to the analytical chemist. He uses this information to devise his analytical scheme. The scene chemist makes mental notes concerning what he believes was the process the operator was using. His sampling scheme is affected by the observations made. Each sample should be geared to address a specific question or questions that will be used to establish that a manufacturing operation was, in fact, taking place at the location. Unless the scene chemist prepares a detailed written report, the information concerning his intuitive impressions of the operation will not be effectively relayed to the analytical chemist.

When the analytical results differ from the on-scene theories, the chemist gains a different perspective of what could have been taking place at the scene. The differing results may address questions the scene chemist had at the scene but could not rectify without a laboratory analysis of the item. The additional knowledge allows the analytic chemist to adapt his analytical scheme and mold his opinions to conform to the new information.

Courtroom presentations should also be considered when addressing how many chemists should be involved. The use of a single chemist provides continuity during courtroom presentations. He can explain the sampling scheme, transition into the laboratory analysis, and finally tie the two together and provide an opinion concerning the operation. All the forensic information can be provided from a single source. The jury receives a less fragmented presentation that walks them through the process. A single chemist addresses what was found at the scene, why samples were taken, and subsequent laboratory results. Finally, as an expert in clandestine manufacturing techniques, he ties all the information together and renders an opinion concerning the totality of the circumstances in the case.

From a case management standpoint, using a single chemist can reduce the overall time necessary to process the samples once they reach the laboratory. As the scene chemist processes the scene, he has subliminally prioritized the samples. Once the samples reach the laboratory, he can analyze only the samples he believes would be necessary to establish the facts of the case. Without a detailed report or specific directions from the scene chemist, the

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analytical chemist is compelled to analyze each sample. This may lead to unnecessary analysis and longer turn-around times for the investigator.

5.1.2 Independent Analytic Chemist

The independent analytic chemist does not have specific knowledge concerning the history of the samples from a clandestine lab operation. Philosophically, it is believed that he will provide objective analytical results. Theoretically, he would not be inclined to skew the analysis to meet the opinions formed at the scene.

The independent analytic chemist does not have independent knowledge of the sample history of the case, and he may be obligated to analyze every sample. Unanswered questions may lead to other problems by not doing so. Assumptions concerning the facts of the case can be avoided by providing the analytic chemist with a detailed report and a complete set of the scene photographs. This information should provide an understanding of the thought process used by the scene chemist at the time the samples were taken. Proper scene documentation should convey this information adequately to avoid as many problems as possible.

The case management philosophy of the forensic laboratory will dictate the use of the scene chemist or an independent analytic chemist to analyze clandestine lab evidence. The proper processing of a clandestine lab scene is a time-consuming process. It can remove a chemist from the bench effectively 1 day or more per scene. The skills required to process a clandestine lab scene are different than those required to analyze the samples. Having chemists trained in specific areas of forensic clandestine lab investigation may provide a more efficient flow of the case through the forensic system.

Documentation and the flow of information are essential to the effective forensic investigation of clandestine lab cases. No matter whether a single chemist or multiple chemists are used, communication is critical. A single chemist must document his activities completely to justify his conclusions at any point during the investigation. A qualified chemist should be able to review the scene chemist's documentation and arrive at the same conclusion. Therefore, providing a copy of this documentation to the analytical chemist should provide the information necessary for him to perform a complete evaluation of the evidence.

5.2 Types of Analysis

The analysis of samples from clandestine labs involves a broader range of analytic techniques than are traditionally used by forensic controlled sub-

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stance chemists. Many of the same instrumental and wet chemical techniques are used. The differences are the way the techniques are applied and the way information is interpreted. Organic and inorganic examinations can be performed on any individual evidentiary sample. Each type of analysis provides insight into the manufacturing process used by the operator. An individual examination type is necessary to establish the identity of a specific chemical used in the manufacturing process, i.e., organic analysis is used to establish the identities of specific precursor chemicals, or inorganic analysis is used to identify reagent chemicals. A combination of the two types of analysis may be required to establish the manufacturing method used, i.e., using a combination of organic and inorganic analysis to establish the presence of the components of a reaction mixture.

The burden of proof required to identify a particular chemical varies with its role in the manufacturing process. Controlled substances have the highest burden of proof, "beyond a reasonable doubt," because their possession is regulated in some manner. The burden of proof for the presence of precursor chemicals varies with the circumstances. The beyond a reasonable doubt standard may apply if possession of the precursor chemical is illegal under a given set of circumstances (e.g., possession with the intent to manufacture a controlled substance). A preponderance of evidence may be all that is required if the chemical's identity is associative evidence, and thus, the burden of proof may be lessened.

The burden of proof determines the level of testing required. Beyond a reasonable doubt requires specific confirmatory tests that will provide a chemical fingerprint of the substance under examination. These fingerprints can be obtained through the use of mass spectroscopy (MS) or infrared (IR) spectroscopy. Techniques such as nuclear magnetic resonance (NMR) and Raman spectroscopy are considered confirmatory tests, but they are not widely available to the forensic chemist analyzing samples for clandestine labs, and they will not be addressed in this chapter.

A series of nonspecific tests indicating the presence of the chemical in question may be sufficient to meet the burden of proof that requires establishing a preponderance of evidence. These examinations can include one or more chemical color tests, microcrystalline tests, or instrumental examinations that produce nonspecific results. The following is an example of a series of nonspecific tests that can be used to establish the identity of a chemical without using a specific test.

Under low-power microscopic examination, a white powder is found to have granules containing a cubic crystalline structure (Test 1). The granules are water soluble (Test 2). A chemical color test indicates the presence of chloride ions (Test 3). A microcrystalline test indicates the presence of sodium ions (Test 4). When combining the information from these four nonspecific

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tests, a chemist could reasonably conclude that the substance is consistent with sodium chloride (NaCl), common table salt.

Techniques such as x-ray diffraction and the use of x-ray detectors could provide specific information concerning the identity of the compound. However, because NaCl is not a controlled substance, the burden of proof does not require that level of detail in the examination. The simple identification of the compound as being "consistent with" NaCl may provide the forensic investigator insight into the manufacturing process used by the operator.

5.2.1 Inorganic Analysis

Many reagent chemicals are considered inorganic (i.e., the molecule does not contain carbon). Their ability to dissolve in water, the resulting pH, and their physical and chemical properties, provide the first insight to their identity. Identifying the inorganic chemicals involved in a clandestine lab or the inorganic components of a reaction and waste mixture enables the clandestine lab chemist to definitively establish the reaction methods that the operator employed or the step in the manufacturing process the operation was in at the time of seizure.

Inorganic analysis is not something routinely performed by the forensic drug chemist. However, many of the same techniques and instruments can be used. The types of tests that can be performed on inorganic compounds include chemical color tests, microscopic examinations, ion chromatography, IR spectroscopy, and the use of x-ray energy.

5.2.1.1 Chemical Color Tests

Chemical color testing is one of the oldest methods of chemical identification. It is a method with which to rapidly establish or exclude the presence of certain categories of compounds or ions. The specificity of the results varies with the test and the ions under examination.

In a chemical color test, a chemical reagent is added to the unknown. The color of the resulting mixture indicates the presence or absence of a group of compounds. For example, a white precipitate resulting from the addition of a 1% solution of silver nitrate to an aqueous solution containing the unknown indicates the presence of chloride ions. Additional testing would be necessary to exclude borate and carbonate ions, which also form a white precipitate with the silver nitrate reagent.

Chemical color tests provide a method with which to identify inorganic acids. The use of a series of three chemical color tests can reveal the identity of a clear acidic liquid. These tests have laboratory and field applications. However, caution should be taken when conducting these tests in the field due to the potentially violent nature of the reactions. Nitric acid is a reagent chemical that reacts violently with certain organic acids and has been known

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Table 5.1 Acid Test Color Reactions

Acid	Silver Nitrate Reagent*	Silver Nitrate + Nitric Acid	Barium Chloride Reagent*	Barium Chloride + Nitric Acid	Diphenylamine Reagent*
Hydrochloric acid (HCl)	White precipitate	Precipitate remains	No reaction	No reaction	No reaction
Hydriodic acid (HI)	Yellow precipitate	Precipitate remains	No reaction	No reaction	No reaction
Sulfuric acid (H_2SO_4)	White precipitate	Precipitate dissolves	White precipitate	Precipitate dissolves	No reaction
Nitric acid (HNO_3)	No reaction	No reaction	No reaction	No reaction	Blue

* See Appendix J for reagent composition.

to cause ignition when it comes in contact with methamphetamine reaction mixtures containing phosphorus.

Simple chemical color tests can be used to quickly provide presumptive information concerning the identity of acidic liquids. The same color test reagents described in Tables 4.4 and 4.5 can be used for the examination of acidic solution under the controlled conditions in a laboratory setting. Correlated in Table 5.1 are the various color reactions with the inorganic acids commonly encountered in clandestine lab operations.

In some laboratories, a simple chemical color test is the only test available to establish the presence of some inorganic compounds (Table 5.2). For example, the reaction between hydrolyzed starch solution and iodide ion produces the characteristic blue color seen in an elementary school science experiment. This may not be a specific identification of iodine. However, to a trained forensic chemist, the color reaction is characteristic enough to

Table 5.2 Color Test Reactions for Inorganic Compounds

Reagent*	Color	Indication
Silver nitrate	White	Cl^-, CO_3^{-2}, SO_3^{-2}
	Crème	Br^-
	Yellow	I^-, PO_4^{-3}
	Brown	OH^-
	Black	S^-
Barium chloride	White	CO_3^{-2}, SO_3^{-2}, SO_4^{-2}, PO_4^{-3}
Diphenylamine	Blue	NO_3^-, ClO_3^-, ClO_4^-, nitro compounds, oxidizers
Thymol	Green	NO_3^-
	Brown	ClO_3^-
Nesslers	Orange	NH_4^+
Starch	Blue	I^-
Sulfuric acid	Yellow orange	ClO_4^-

* See Appendix J for reagent composition.

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establish the preponderance of evidence of its existence. The chemist cannot make a statement concerning the existence of iodine in a sample without testing to support his conclusion. This simple color test provides that support for those situations in which the laboratory does not have access to the instrumentation that can establish the presence of iodine beyond a reasonable doubt.

5.2.1.2 Microscopic Techniques

Microscopic examinations of inorganic compounds are the second type of testing that can be performed on inorganic compounds. As in chemical color testing, the specificity of the results depends upon the compounds being examined and the tests being performed.

The three types of microscopic examination involve observation of the compound's basic optical properties, recrystallizations, and microcrystal examinations. Each type of microscopic examination requires different levels of microscopic expertise. Each method requires practice on the part of the examiner to be able to recognize crystal structures as specific to a given ion.

Observing the optical properties of pure compounds under the microscope can be used to identify them (Table 5.3). Information concerning the compound's color, crystal form, and index of refraction can be used to make a specific identification. Contained in Appendix G is a table of the optical properties of inorganic compounds found in clandestine laboratories. The physical structures and optical properties of a compound or a mixture of compounds can be observed by placing the unknown in a drop of nonvolatile organic liquid, such as mineral oil, or the Cargile liquids that are used to establish the refractive index. The use of polarized light and optical filters can assist the chemist in visualizing the various crystals as well as can provide information concerning their birefringence and other optical properties.

Recrystallization is a method in which the inorganic components of pyrotechnics have been identified. The component is dissolved in a minimal

Table 5.3 Microcrystal Development Techniques

- Add a few crystals of the unknown to a liquid in which the substance IS NOT soluble, and observe the crystalline structure and optical properties.
- Dissolve a few crystals of the unknown in a liquid, and observe the crystals that develop as the liquid evaporates.
- A drop of reagent solution is caused to flow into the test drop.
- A drop of the reagent is added directly to the test drop at the center (or vice versa).
- The reagent and test drop mixture is scratched or mixed to induce crystal formation.
- Reactions take place in a capillary tube.
- A fragment of solid reagent is added to the test drop.
- A drop of the reagent is suspended over a test drop (or vice versa).
- A drop of acid, base, or solvent may be added to the test drop to assist in the volatilization.

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amount of deionized water and is placed under a microscope. As the water evaporates, the solution reaches the saturation point, and the compound begins to crystallize around the edge of the drop. The shape of the resulting crystals is characteristic of the compounds in the sample. As with other microscopic techniques, the use of a polarized light microscope is beneficial but not absolutely necessary.

Microcrystal tests are conducted in a manner similar to how chemical color tests are conducted. A chemical reagent is added to the substance under examination. Instead of observing the resulting color, the examiner looks for the formation of characteristic crystals under the microscope. The use of a polarized light microscope is not necessary but can be beneficial. Caution should be exercised, because a number of anions may produce similar crystals using the same chemical reagent. However, results of a series of microcrystal tests can be considered specific identification for an anion. Listed in Appendix H are the various reagents used for inorganic microcrystal examinations.

Microcrystal tests can be used on pure compounds as well as mixtures. There is a limitation. A single test will only identify one half of the inorganic compound. Only the cation (Y^+) or the anion (X^-) component is identified using a given reagent. Additional tests need to be undertaken to identify the other half of the compound. In mixtures, this can be problematic if there is more than one set of cations and anions present. The burden is on the examiner to establish which cation is paired with which anion. Assumptions can be made using information from the scene. However, if the only information the analytic chemist has is the sample in front of him, he may be hard pressed to "prove beyond a reasonable doubt" what the cation and anion pairing was originally.

The use of microscopic techniques as a means of identification can be problematic if the testing is not documented. All testing used to make identifications should be documented in such a manner as to allow an independent expert to evaluate the results. Instrumental techniques, such as MS and IR spectroscopy, provide a paper record of the results of the examination. This demonstrates that the analytic chemist actually performed the test, documents the results, and provides a means of independent evaluation if that becomes necessary.

When the analytic chemist utilizes microscopic techniques as a means of identification, he should follow the same protocol of documenting his test results, as he is obligated to do when utilizing instrumental techniques. It is recommended that the results of microscopic examinations used to identify compounds be documented through the use of photomicrographs. This provides a record of examination used to identify the compound. This also provides a means for an independent evaluation of the results. If he cannot use photomicrographs to document his examination, he should sketch and

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describe the crystal forms he observed and used to make his identification (Table 5.4). Each photomicrograph or sketch should have documentation correlating the resulting crystal form to the sample preparation technique used.

5.2.1.3 Infrared Spectroscopy

Infrared (IR) spectroscopy has long been used as a method of positively identifying organic compounds. A compound's IR spectrum has been called its chemical fingerprint. For the identification of inorganic compounds, it has not been extensively used in the forensic arena. This may be due to the broad bands and lack of detail in the spectra. Even with these handicaps, IR spectroscopy can be used to identify inorganic compounds.

The two keys to using IR for inorganic compound identification include sample preparation and peak identification. As with any analytical technique, sample preparation is the key to obtaining a usable spectrum. The exact locations of peaks in the IR spectrum are critical in identifying the salt form of an inorganic compound.

The broad absorbance bands of inorganic IR spectra make it difficult to identify the maximum absorbance of the peak. The sample concentration should be diluted until a definite peak is observed in the primary absorbance band. This will allow the examiner to identify the maximum absorbance of each peak of the spectrum.

Determination of the maximum absorbance of each peak in the spectrum is critical. A shift in the maximum absorbance of 10 wave numbers can be the difference between the sodium and potassium salt of a compound. These minor shifts may seem insignificant, but if they are reproducible in properly prepared samples, they provide the specificity required for identification purposes.

Sample preparation is critical when using IR to identify and differentiate inorganic compounds. Many inorganic compounds are efficient absorbers of IR radiation and easily overload the test sample. It is essential that the primary absorbance bands of the resulting IR spectra have well-defined peaks with resolvable maximum absorbance values, as opposed to broad nondescript bands with rounded or flat maximum absorbance areas. Broad rounded absorbance bands do not show subtle absorbance shifts needed to differentiate between salt forms of an inorganic compound.

Many anions have characteristic absorbance bands in the IR spectrum. These can be used as a screening tool to classify the type of inorganic compound with which the examiner is dealing. The presence of particular anion absorbance bands in a mixture can provide information that can be used to categorize the types of compounds that may be present. Appendix I contains a table of anions and their corresponding IR absorption wavelengths.

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Table 5.4 Crystal Descriptions

Crystal	Shape	Description
Blade		Broad needle
Bunch / Bundle		Cluster with the majority of the crystals lying in one direction
Burr / Hedgehog		Rosette, which is so dense that only the tops of the needles show
Cluster		Loose complex of crystals
Cross		Single cruciform crystal
Dendrites		Multibrachiate branching crystals
Grains		Small lenticular crystals
Needles		Long thin crystals with pointed ends
Plates		Crystals with the length and width that are of the same magnitude
Prisms		Thick tablet
Rod		Long thin crystals with square cut ends
Rosette		Collection of crystals radiating from a single point
Sheaf		Double tuff
Splinters		Small irregular rods and needles
Star		Rosette with 4 or 6 components
Tablet		Plates with appreciable thickness
Tuff / Fan		Sector of a rosette

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In some instances, the results from IR analysis cannot distinguish between salt forms of a given compound. In these situations, the analytic chemist may be able to use the results of other techniques to make a specific identification. For example, the IR spectra of sodium and potassium cyanide are almost indistinguishable. However, sodium and potassium are easily distinguishable using microcrystal techniques. Combining the results of these examinations provides the analytic chemist with the information necessary to render an informed opinion.

5.2.1.4 Ion Chromatography

Ion chromatography (IC) is an instrumental method that allows the chemist to identify the anion and the cation of an inorganic substance or mixture. The analysis is a three-step process. First, the cation is determined. Then, the anion is determined. Finally, the results are combined, and the compound is determined. (e.g., $Na^+ + Cl^- \rightarrow NaCl$). Additional testing may be necessary to establish the hydration state of a compound that contains multiple hydration states.

Ion chromatography is effective in separating and identifying cations and anions. However, when there are multiple components in a solution, the IC cannot distinguish which cation is associated with what anion, or what were the forms of the compounds originally placed into the mixture. Here is where the chemist uses his knowledge of clandestine manufacturing methods, along with the chemical inventory from the clandestine lab scene, to establish the most probable combination of chemicals that would produce the results obtained from an IC run on a complex mixture.

An example of how ion chromatography can be used to propose a reaction mechanism would be the analysis of a basic aqueous solution from a clandestine lab that contained a trace of methamphetamine. Anion analysis revealed the presence of iodide with small amounts of chloride and carbonate present. Cation analysis revealed the presence of sodium. From this information, the chemist proposed that the iodide originated from HI, and the chloride originated from the HCl salt of the ephedrine precursor. The sodium came from sodium hydroxide that was used to neutralize the HI. The odd trace of carbonate was a result of the sodium hydroxide reacting with the carbon dioxide in the air to produce sodium bicarbonate.

5.2.1.5 X-Ray Analysis

The use of the x-ray detector on various instruments provides the analytic chemist with a method for identifying the elemental composition of a compound or mixture. This is accomplished by recording the energy emitted from the substance that has been exposed to a beam of electrons. Each element releases a characteristic wavelength(s) of x-ray energy, as it releases

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the energy it absorbed from the electron beam. The instrument also calculates the percentage of each element in a sample. This information can be used to determine the molecular formulas of most inorganic compounds.

The drawback to this method is that many x-ray detectors cannot detect hydrogen, carbon, nitrogen, and oxygen. This limits their use in organic analysis and in the determination of hydrogen, oxygen, nitrogen, and certain low-molecular-weight metals, e.g., lithium that can be used in the Birch reduction method is outside the detectable range of most x-ray detectors.

Mineral acids are an example of compounds that cannot be directly identified using x-ray technology. Besides the acid being corrosive and detrimental to internal instrument parts, the detector cannot detect hydrogen. However, derivatization techniques can be used to compensate for this problem. The chemist replaces the undetectable H with a detectable element, like potassium or sodium, by reacting the mineral acid with a strong base. The water is evaporated, and the remaining solid is analyzed (e.g., $HI + NaOH \rightarrow NaI + HOH$).

X-ray techniques can be used to analyze solid inorganic waste to determine the elemental makeup of the mixture. The dried waste solids are essentially the same inert inorganic salts that are created when a mineral acid is neutralized with a strong base. However, these waste solids can contain other inorganic by-products that may complicate data interpretation. A Practical Example in Chapter 9 demonstrates the utilization of this technique.

5.2.2 Organic Analysis

The examination of organic compounds is the analysis that is most familiar to the forensic chemist who analyzes controlled substances. The methodology and instrumentation used to analyze drugs of abuse and explosives are routinely used in the analysis of organic compounds. Gas chromatography (GC), IR spectroscopy, MS, chemical color, and microscopic techniques can be used to analyze organic compounds. As in inorganic analysis, each technique has advantages and limitations.

5.2.2.1 Test Specificity

The analysis of individual organic chemicals is relatively straightforward. The process begins with a visual examination, followed by presumptive testing, and concluding with a confirmatory examination. However, before this process can commence, the analytic chemist must decide what degree of certainty is required for the identification of this exhibit. The degree of specificity needed to identify a solvent may be different than that needed to identify a precursor chemical or a controlled substance. The degree of specificity needed will determine the type of examination sequence used to identify the compound.

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Solvents are an example of chemicals that have a low degree of specificity needed for their identification. Positive identification of the type of solvent used is generally unnecessary to establish the elements of a manufacturing charge. The solvent's identity only assists the chemist in formulating his opinion on the manufacturing method being used. Generally, the scene chemist can establish the probable identity of a solvent by comparing a container's contents to the label. If the contents look, feel, smell, taste, and act like the solvent on the label, generally, that will be sufficient to establish the probable identity of the solvent. If it does not react as expected, the scene chemist should perform presumptive tests on the substance to confirm or refute the label, and possibly sample the substance for laboratory analysis.

Clear liquid solvents present a unique problem. Laboratory analysis is the only way to determine whether a controlled substance is dissolved in them. What appears to be an unused liquid may, in fact, contain the final product or other components that would give light as to the manufacturing process used. Therefore, at a minimum, the analytic chemist should perform a screening examination on unknown solvents and not disregard them just because the liquid sample appears to be unaltered.

Reagents are an example of chemicals that require a moderate degree of certainty in establishing their identity. Reagents are not controlled, but they are used to create a controlled substance. Their identification may help in identifying the manufacturing route. An example of using a reagent chemical's identity to establish a manufacturing method would be the use of acetates to manufacture phenylacetone. Sodium and lead acetate can be used to manufacture phenylacetone. Each is used in a specific synthesis route, and they are not interchangeable. A simple microcrystal test for the presence of sodium or lead, along with a chemical color test for the presence of an acetate, can be used to identify the type of acetate, which helps identify the manufacturing route the operator probably used.

Nonspecific presumptive tests include chemical color and microscopic techniques, ultraviolet (UV) absorbance, GC retention time, index of refraction, or density. Each test has its own degree of specificity. A combination of these techniques is necessary to rule out other compounds.

Precursor chemicals and the controlled substances they form, as a rule, need to be specifically identified. This usually involves using instrumental analysis to confirm any presumptive test that may have been done. The possession of precursor chemicals may not be controlled. However, the necessity of their positive identification increases when they are possessed in conjunction with the appropriate reagent chemicals or equipment, which creates the potential ability to produce a controlled substance. In that situation, the identity of the precursor chemical should be specifically established, as well as the identity of any controlled substance.

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The specific tests used in most forensic laboratories include IR spectroscopy and MS. NMR and Raman spectroscopy are also considered specific tests; however, this book will not address those techniques. Each technique is considered specific for the identification of a compound, but each has its limitations. For example, the salt form of a compound cannot be determined by using MS. With MS, there may also be problems differentiating between stereo- and geometric isomers. The resulting ion patterns can be almost indistinguishable. Without retention time data or derivatization before analysis, identification is not completely possible with MS alone. IR spectroscopy cannot distinguish between optical isomers.

With a significant portion of the samples submitted for laboratory analysis, it is required that organic compounds be identified. The composition of the samples may vary, but the procedure remains the same. Each sample requires a screening step, an extraction or sample preparation step, and a confirmatory step. These steps can be subdivided into wet chemical or instrumental procedures. Wet chemical procedures are used as screening methods or for sample preparation. Instrumental procedures are used for screening or as a confirmation tool.

5.2.3 Wet Chemical Procedures

Wet chemical procedures are used in the initial stages of the organic chemical identification process. These nonspecific tests provide a method with which to quickly indicate whether a controlled substance is present within a sample. These procedures can also be used to isolate controlled substances for confirmatory testing using instrumental techniques. Wet chemical procedures consist of chemical color tests, microscopic techniques, thin layer chromatography, and various extraction techniques. A series of these tests can be used to deductively identify a compound or mixture.

5.2.3.1 Chemical Color Tests

Chemical color tests are chemical reactions that provide information regarding the structure of the substance being tested. Certain compounds or classes of compounds produce distinct colors when brought into contact with various chemical reagents. (See Appendix J for a list of color test reagents and their compositions.) These simple reactions can indicate the presence of generic classes of compounds.

Chemical color tests are generally conducted by transferring a small amount of the substance being tested to the well of a spot plate or into a test tube. The test reagent is added to the substance. Some tests may be conducted in a sequential fashion utilizing multiple reagents. The results of each step in the sequence are observed and noted. Positive and negative controls should be run on a regular basis to ensure the reliability of the testing reagents.

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There is a certain amount of subjectivity when a color is reported. It is not uncommon for two people to describe the same color differently. The colors produced can also be influenced by the concentration of the sample, the presence of diluents and adulterants, and by the age of the reagent. The length of time the reaction is observed may also influence the color reported. Color transitions and instabilities are not unusual. Allowances should be made for these differences.

5.2.3.2 Microscopic Techniques

Microscopic techniques are used as a screening tool to confirm a diagnosis made using other testing methods. Many of the same microscopic techniques used for inorganic analysis have organic applications as well. They are fast, simple to administer, and can be highly specific. There is a debate as to whether they are specific enough to be used as a confirmatory test.

The microscopic crystal structures of a compound can be used to tentatively identify components within a solid mixture. The examiner can obtain a profile of the various components within the mixture by placing a sample into a liquid test drop in which most, if not all, of the components are insoluble. (Mineral oil works well for this type of analysis.) The component's physical and optical characteristics are then observed under plain or polarized light. Commonly encountered components can be tentatively identified and quantitated when using this technique.

Microcrystal tests involve observing the crystals formed when the questioned sample is reacted with a test reagent. The test reagent and the sample can be combined using any of methods described in Table 5.3. A reaction between the component of interest and the test reagent forms a solid compound that is not soluble in the test drop. This solid forms uniquely shaped crystals that can be observed with a microscope (Table 5.4).

Microcrystal tests can also be used to determine the optical isomer of a compound. Single isomer compounds (*d* or *l*) produce different crystal forms than a racemic mixture (*d* and *l*) of the same compound. Single isomer crystals will form if a substance with the same isomer is added to the test solution prior to addition of the test reagent. Racemic crystals will form if the opposite isomer configuration is added to the test solution prior to analysis.

Mixed crystal examinations can give insight into a compound's optical orientation. They are performed by seeding a sample with a known isomer of the substance under examination. The crystals that result from the addition of reference material with the same optical orientation will result in single isomer crystals. Racemic crystals are formed if the optical orientation of the two compounds is different.

Microcrystal identification relies on the comparison of the crystals formed by the unknown with those formed by a reference standard using

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the same reagent. Difficulties obtaining a match between the crystals of the unknown and those of the reference sample may arise. Impurities in the unknown sample may lead to the formation of deformed, irregular, or unusual crystals. These problems can be overcome by utilizing a cleanup procedure, such as TLC, extractions, or particle picking, prior to microcrystal analysis.

Polymorphism can occasionally be a source of trouble. Sample concentration and reagent age can lead to the creation of different microcrystalline forms. This reemphasizes the comparative nature of microcrystal identification. The comparison should be done using the same sample concentration with the same crystal reagent.

Differences in crystal appearance can arise from the concentration of the solution. The crystals in highly concentrated test drops develop rapidly, resulting in a distortion of the classic crystal shapes. Concentrated test drops should be diluted to a concentration that produces classic crystal forms that are conducive to comparison and identification.

The reagent's age will also affect crystal development. Therefore, unknown and reference samples should be run using the same reagents, under the same conditions, and at approximately the same concentrations. Reagent should also be checked on a regular basis to ensure not only that it will produce crystals with reference standards, but also that the crystals produced are consistent with the accepted crystal form for the reaction between the reagent and the substance in question.

5.2.3.3 Thin-Layer Chromatography

Thin-layer chromatography (TLC) is a wet chemical test used to screen for the presence of drugs and explosives. It is a separation technique that utilizes molecular mobility and solvent compatibility to separate and distinguish compounds within a mixture. In other words, the way a component dissolves in the TLC solvent and how it reacts with the coating on the thin-layer plate as the solvent travels over it affects the separation. Compounds are separated by their size, shape, and reactivity with the solvent, similar to rocks flowing down a river. Small compact molecules will travel across the TLC plate at different rates than large rambling molecules.

In the typical TLC procedure, a sample of the unknown is placed toward the bottom of a glass plate containing a thin layer of silica gel. A sample of a reference compound is placed the same distance from the bottom of the plate. The TLC plate is placed into a tank containing a solvent (or mixture of solvents). As the solvent travels up the TLC plate, the various components within the sample are separated. When the solvent migration is stopped, the TLC plate is removed from the tank, and the solvent is allowed to evaporate. The compound movement is then visualized through observation under UV

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light or through development with a chemical color reagent designed to react with various compounds.

The *Rf* value is used to establish the identity of the spots on the TLC plate. The use of *Rf* values for a known solvent system only provides a generic insight as to the identity of the unknown spot. They should not be relied upon for confirmation of unknowns. A known reference sample, run on the same TLC plate, should be used for comparison.

Rf values can be affected by many factors. The adsorbent uniformity on the thin-layer plate, sample concentration (spotting is too weak or strong), room temperature during the mobile phase, and development distance of the solvent during the mobile phase, all will affect the results. Care should be taken to eliminate variances in the method caused by any of these factors. Placing a reference sample containing the suspected compound on the TLC plate with the questioned sample reduces the variables involved in TLC comparisons.

5.2.3.4 Extractions

Extractions are not a screening test per se. However, the fact that the compound was isolated as a result of the extraction indicates that the compound had certain chemical characteristics. These are class characteristics the can be used to deductively support the confirmatory test.

Extractions are used to separate the compound of interest from the rest of the sample. The type of extraction used will depend upon the compound of interest and the matrix in which the compound is located. In some cases, multiple extraction techniques are necessary to separate the substance of interest from the remainder of the sample. In other instances, instrumental analysis is the only way to separate compounds with similar chemical properties for confirmation.

The screening techniques used should be designed to identify as many of the components of the sample matrix as possible. This allows the examiner to select the extraction technique that efficiently and effectively isolates the component of interest from the rest of the compounds. Misidentified or unidentified components within a sample mixture may lead to the selection of an inappropriate extraction technique, which in turn, may affect the results of the confirmatory test.

5.2.3.5 Wet Chemical Documentation

Wet chemical tests are generally nondocumentable techniques. There is no independent record of the performance of the test. The test documentation solely rests on the examiner's handwritten notes. Therefore, the chemist should describe his observations as completely as possible. A (+) or (–) notation next to a test name does not provide a peer reviewer insight as to the examiner's observations during the performance of the test.

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The colors or transition of colors that were observed during the course of a chemical color test should be described. Photographing a chemical color test may or may not be a solution to the documentation issue. Photography demonstrates the color that was observed during the examination. However, it may only preserve a portion of the test. Many chemical color tests have a transition of colors from the beginning of the test to the end. Photographs do not adequately reflect the examiner's total observations.

No supporting documentation is generally generated with microcrystal examinations. Therefore, the examiner's description of his observations should be as complete and accurate as possible. When definite crystals are formed, their forms and habits should be noted (described, sketched, or photographed). Listed in Table 5.4 are descriptive terms with diagrams that can be used to describe the observed crystals.

Lack of supporting documentation may be less significant if microcrystal tests are used as a screening tool. However, if they are to be used as a tool to specifically establish a compound's identity or isomer configuration, steps should be taken to provide reliable documentation concerning the examiner's observations. Photomicrographs should be taken of the microcrystals that were used to make identifications. The photomicrographs should be included in the examiner's notes for peer review, when necessary.

As with chemical color and microcrystal examinations, no supporting documentation is generally generated using TLC. Accurate notes regarding the solvent system used should be included in case notes, along with the *Rf* calculations used for compound identification. Any deviations from the referenced method or unusual occurrences should also be documented. The examiner should thoroughly describe the observations used to make his conclusions, including the colors and patterns observed on the TLC plates as well as any observations made under UV light.

Photography of TLC plates is an option. Photographs can document the examiner's observations of the colors and positions of the sample spots. If the photograph is scaled properly, a peer reviewer or independent examiner can calculate *Rf* values.

The extraction phase of the analysis is not used for preliminary or confirmatory identification purposes. However, it is a means to those ends. As such, it should be documented. Peer reviewers should be able to evaluate the extraction technique used to prepare the sample for any subsequent testing.

5.2.4 Instrumental Examinations

Instrumental examinations are documentable testing methods. This point is key to the confirmation process. It is not enough for the examiner to be able to say the compound had the same chemical fingerprint as the substance in question. He has to be able to demonstrate it beyond a reasonable doubt.

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This includes subjecting the examination to peer review. Instrumental examinations provide the vehicle for this review.

There are four basic instruments routinely utilized by forensic chemists analyzing clandestine lab samples. The UV spectrophotometer and the GC are used as screening and quantitative tools. Liquid chromatography is utilized as a screening tool but not as widely. The IR spectrometer and the mass spectrometer are instruments used to confirm the identity of unknowns. As stated previously, NMR and Raman spectroscopy are used as confirmatory tools, but they do not have as broad a base of use.

5.2.4.1 Ultraviolet Spectroscopy

Ultraviolet (UV) spectroscopy is an instrumental technique that provides compound classification. It is a screening tool and not a confirmatory test. Although some compounds exhibit unique UV spectra, the spectra are considered class characteristics and do not contain sufficient detail (individual characteristics) to be considered a compound's chemical fingerprint.

The two general uses for UV spectroscopy in the controlled substances unit are general screening and quantitation. The shape of the spectrum provides insight into the identity of the compound. The amount of UV light absorbed can correlate to the amount of substance in the sample.

UV spectroscopy is a useful tool for single-component analysis of samples with known or suspected composition, such as pharmaceuticals. The UV spectrum can confirm or rebuff the composition of the preparation under examination. However, if compound identification is required, it should be done using a specific test such as IR or MS.

Mixtures of compounds capable of absorbing UV energy can present an analytical problem. Compounds have differing capacities to absorb UV light. A strong UV-absorbing substance mixed with a controlled substance that is a weak UV absorber, may result in a UV spectrum that does not reflect the presence of the controlled substance.

Quantitation is another venue in which UV spectroscopy is useful. To be most effective, the sample should contain a single UV-absorbing component. If there are multiple UV absorbers in the sample, the component of interest should have a distinct resolvable absorption band. The quantitation procedure can be as simple as comparing the concentration of the suspected tampered sample with that of a known unaltered sample. The UV absorbances should be the same if the concentrations and compositions of the samples are identical. An in-depth analysis can determine the actual concentration of the substance in question. The absorbance value of the test sample is compared to the absorbances of a series of known solutions. The concentration of the test sample can be taken from the graph of concentration versus absorbance values of the reference samples.

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5.2.4.2 Gas Chromatography

Gas chromatography (GC) is a documentable chromatography form that can be used in lieu of TLC. It is not a specific confirmatory test for controlled substances. However, dual-column techniques and the evaluation of alkaloid peak patterns can be used for identification purposes. The GC is also used as a separation device for confirmatory examinations, such as MS and Fourier transform IR spectroscopy (FTIR).

The GC separates compounds by their size, shape, and reactivity with the chemical coating of the GC column, in a manner similar to rocks flowing down a river. The carrier gas acts as the water, and the column coating acts as the riverbed. The small molecules travel through the chromatographic column more rapidly than larger molecules. Their shapes and their reactivities with the column's coating separate molecules of the same size.

Chromatograms from GCs are used to identify unknowns based on the retention time or relative retention time of a peak under certain operating conditions. The retention time (*Rt*) is the time it takes a compound to travel from the injection port of the GC to the detector. The relative retention time (*RRt*) is the ratio of the retention time of the substance to the retention time of an internal standard placed into the sample.

The *RRt* is considered a more reliable value. The use of an internal standard provides a reference point with which to calculate *RRt* values. It also demonstrates the precision and accuracy of the instrument. The internal standard eluting at the proper time indicates that the gas flow and oven conditions are operating properly. The size of its peak indicates proper operation of the detector, if the concentration of the internal standard is known.

The GC can be used to differentiate geometric isomers. An example of the use of GC retention times to differentiate between isomers is the identification of the *cis*- and *trans*-phenylaziradines that are by-products of the HI reduction of ephedrine to methamphetamine. Even though these compounds have essentially the same mass spectrum, the GC retention times are significantly different. On a nonpolar GC column, the *cis*- isomer has a retention time noticeably less than the *trans*- isomer. Baseline resolution of the three isomers of the explosive compound dinitrotoluene is another example.

Analysis by GC alone is not generally considered confirmation of a controlled substance. More than one compound could possibly have a given *Rt* or *RRt*. Therefore, with conventional detectors (i.e., flame ionization, electron capture, nitrogen/phosphorus, etc.), the chemist cannot definitively tell what compound elutes at a given *Rt* or *RRt*. The specificity increases with the specificity of the detector. For example, the use of a nitrogen/phosphorus detector will only detect compounds containing nitrogen or phosphorus, thus, narrowing the field of potential organic compounds. Fortunately, this group is the one to which many drugs and explosives belong.

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Dual-column GC has been used as a confirmatory test. A single sample is injected into a GC that divides the sample into two chromatographic columns. Each column contains a different liquid phase (the interior coating that causes compound separation). A compound is considered identified if it has the proper *Rt* or *RRt* values on both columns.

Commonly, GCs are used as separation tools for the confirmatory tests of MS and FTIR. The GC separates the compounds, and the MS or the FTIR provides information concerning the chemical properties of each of the compounds as they elute from the chromatographic column.

Quantitation is another use for the GC. This can be accomplished by analyzing a series of diluted samples using a method similar to that used in UV analysis. The other method uses the relative response of the item in question to that of an internal standard.

As a quantitation tool, GC has an advantage over UV. The effects of multiple components within the sample are reduced or eliminated, because the GC separates the components of the sample during the analysis. The compound's UV absorbtivity also does not affect the analysis. Each component has a similar detectability range with a given detector.

5.2.4.3 Mass Spectroscopy

Mass spectroscopy (MS) is the workhorse instrument used by the forensic chemist. It uses the pattern of molecular pieces (ions) produced when a molecule breaks apart after it is exposed to a beam of electrons as a means of identification. The resulting characteristic pattern is called the mass spectrum. It is considered one of a compound's chemical fingerprints.

The mass spectrometer exposes the compound under analysis to a beam of high-energy electrons that shatters the molecules. The mass spectrometer then sorts and counts the resulting pieces (ions) and produces a pattern, the mass spectrum. When the energy of the electron beam remains constant, the molecule will produce the same mass spectrum, which is considered one of the compound's chemical fingerprints.

MS has its limitations. It cannot differentiate among certain types of isomers. Stereoisomers and geometric isomers may produce mass spectra that are essentially identical. Stereoisomers (molecules that are mirror images of each other) have identical mass spectra. Without additional information, i.e., GC retention time data, the chemist may not be able to say the compound was one isomer or the other. Ephedrine and pseudoephedrine are examples of two compounds that have essentially the same GC retention times and mass spectra.

Geometric or positional isomers will also produce similar, if not the same, mass spectra. Many times, the compounds can be differentiated by their chromatographic retention times. Other times, there are one or two clusters

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of ions that have ratios specific to a particular isomer. Methamphetamine and phentermine are two geometric isomers that can be differentiated through the use of MS.

The mass spectrometer generally cannot distinguish between the salt- and freebase form of a drug. The salt portion of the compound is generally outside the detection range of the MS. The detector only "sees" the freebase portion of the compound.

The information obtained from the mass spectrometer can be used to establish the synthesis route used to manufacture the controlled substance. Each reaction produces by-products. In some instances, the by-products produced are specific to a particular manufacturing method. Even if the detected by-products are not specific to a reaction, their presence can be used to corroborate other information, i.e., notes, chemicals on hand, etc., as to the method of manufacture. Shown in Appendix K is a five-peak table of drug precursor chemicals, controlled substances, and by-products. Also included is the reaction indicated by the presence of these compounds.

There are a number of mass spectra libraries available to assist in the identification of unknowns. The spectra in these libraries can provide insight into the identity of numerous components that can potentially be within these mixtures. However, final confirmation is only accomplished by comparing the mass spectra of the unknown to the mass spectra of a traceable reference standard. The reference spectra should be obtained on the same instrument, under the same operating conditions. The burden of proof required in a given situation will dictate how the information from these libraries should be used.

Library spectra should not be used for proof beyond a reasonable doubt. The variations in operating parameters between the instrument used to obtain the sample's spectrum and the one used to obtain the library spectra will differ. These deviations may be subtle, but they can be significant enough to eliminate the compound as the source.

Library spectra provide a preponderance of evidence concerning the identity of a compound. Many of the by-products of clandestinely produced controlled substances do not have traceable primary standards that can be used for positive identification purposes. However, their probable identity, established through a mass spectral library search, can be used as associative evidence to render opinions concerning the manufacturing methods used in the operation.

5.2.4.4 Infrared Spectroscopy

Infrared (IR) spectroscopy has been the traditional method used for confirming the identity of a controlled substance. Traditionally, the sample went through a series of screening tests to establish the compound's suspected

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identity, and the identity of any adulterants or diluents were determined. The controlled substance was then extracted and purified. Finally, an IR spectrum was obtained. With the instrumentation of modern technology, an IR spectrum can be obtained from a single particle or from a peak in a GC run. This has reduced the need for the nonspecific tests used as screening tools and the extractions necessary to isolate the compound of interest.

IR spectroscopy uses a compound's ability to absorb IR light as a means of identification. The bond of each of the molecule's functional groups will absorb specific wavelengths of IR radiation. The exact wavelength will depend on the arrangement of the functional groups on the molecule. The pattern that results from charting the absorbance or transmittance of IR light that is pasted through (or reflected from) a sample is considered a chemical fingerprint.

The ability to differentiate between isomers is a benefit of using IR as a confirmation tool. Compounds with isomers that are indistinguishable by MS may be differentiated through the use of IR. The position of the functional groups on the molecule dictates how they will vibrate, which affects the wavelength of IR radiation that is absorbed. The stereoisomer of the compound may allow or hinder the vibration of a particular functional group. This allows the chemist to differentiate between stereoisomers such as ephedrine and pseudoephedrine (Figure 5.1). However, optical isomers, i.e., *d*-ephedrine and *l*-ephedrine, do not exhibit significant differences in their IR spectra.

In some jurisdictions, a compound's salt form may be important in determining the sentence after a conviction is obtained. IR spectroscopy can be used to identify a compound's salt form. In Figure 5.2, a differentiation between freebase cocaine (crack) and cocaine hydrochloride is made. The specific salt form can be used to establish a manufacturing method. Shown in Figure 5.3 are the IR spectra of the HI and HCl forms of methamphetamine. In both examples, the most obvious difference is demonstrated in the spectra's front portion (4000 cm^{-1} to 2000 cm^{-1}).

IR spectroscopy is also useful in differentiating structural and geometric isomers that the MS cannot without derivatization, retention time data, or both. Changing the position of a functional group on an aromatic ring will change the IR spectrum enough to allow easy identification. Figure 5.4 is an example of the IR differences of the three structural isomers of dinitrotoluene. The only difference is the position of one nitro ($–NO_2$) group on the aromatic ring.

Traditionally, IR confirmation has been limited to compounds that have gone through some type of extraction to produce a pure compound prior to analysis. Analysis time could be lengthy, depending on the resolution the analyst desired. Advances in technology have reduced the time required for

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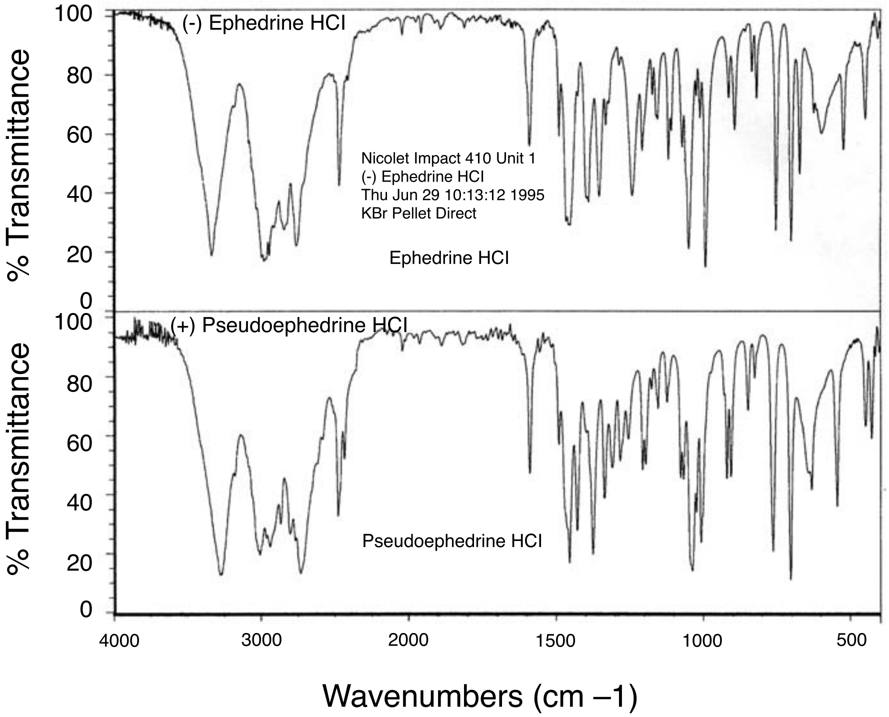

Figure 5.1 Ephedrine/Pseudoephedrine IRs.

sample preparation and analysis. With the advent of FTIR analysis, time has gone from minutes to seconds. The ability to obtain an IR spectrum instantaneously has allowed FTIR detectors to be used in conjunction with GCs. This allows the chemist to identify the components of a mixture by IR without first separating each of the components. Additionally, the micro-FTIR can isolate and obtain IR spectra of individual particles within a mixture.

Sample preparation is a key element in IR examinations. The physical state of the sample will significantly affect the resulting spectra. For example, the spectra obtained from the GC/FTIR will be in the vapor phase. These spectra will be different than the liquid- or solid-phase IR spectra a chemist traditionally uses for identification purposes. Pellet spectra of solid samples will vary from those produced using the thin-film technique. Transmission spectra and reflectance spectra of the same compound will have variations. There can even be significant variation between thin-film spectra of the same compound that are a result of polymorphism when the compound crystallizes. Each sample preparation technique produces a unique reproducible result that can be used for identification purposes. However, the analytical chemist must be sure to compare “apples to apples” when making an identification.

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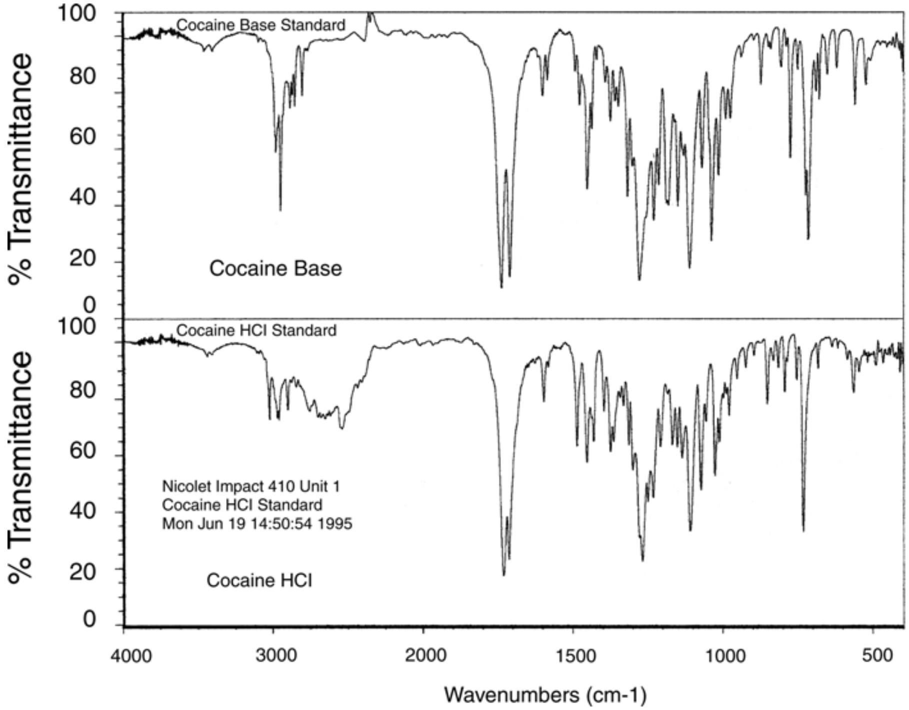

Figure 5.2 Cocaine Base/Cocaine HCl IR comparison.

With computerization, we now have the ability to compare the IR spectra of an unknown to those in various libraries. As with the MS library searches, the results should not be used for identification purposes for compounds that require proof beyond a reasonable doubt. The compound's physical state, type of detector used, and sample preparation techniques will affect the spectra obtained and the results of a computerized library search. Identification should only be made by comparing the spectra from the questioned sample to the spectra of a sample from a traceable reference that was prepared under the same conditions, using the same instrument.

5.2.4.5 Documentation

Instrumental techniques are documentable in that they generate analytical data in a form that demonstrates that the analysis was performed. The data are objective and can be subjected to peer review as part of a quality assurance program or independent evaluation at a later date. Interpretation of this data is less subjective than in other areas of the forensic laboratory. However, it is still subject to interpretation.

For peer review purposes, case notes or instrument printouts should include the operating conditions of the instrument during the analysis. This

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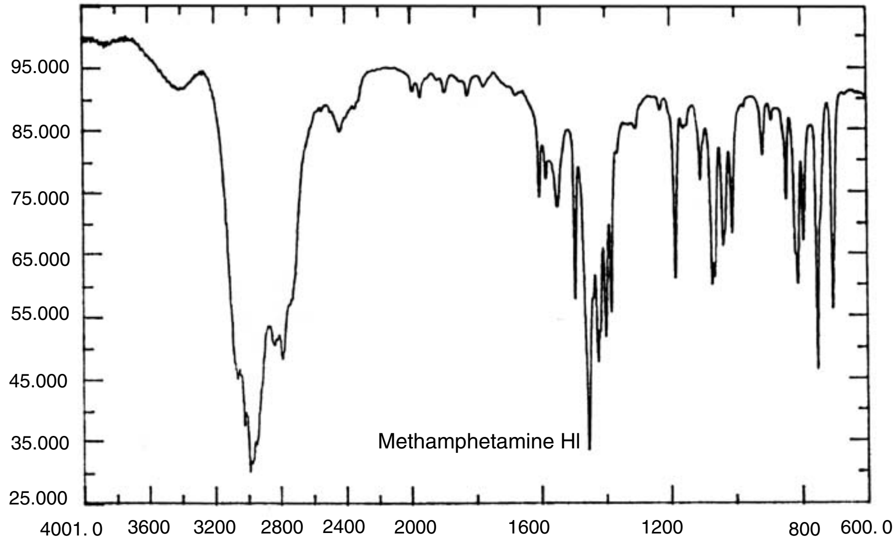
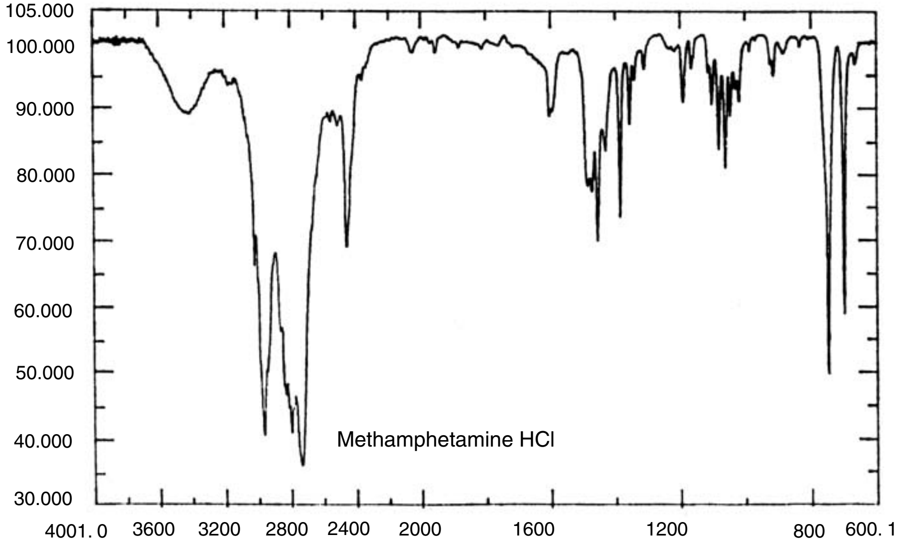


FN : C TITLE : METHAMPHETAMINE HI
DS1 : HOH EXT / ETHER WASH / CHCl 3 EXT / ACETONE EXT
DS2 : THIN FILM ON KBR PLATE
X =WN Y=%T DATE = Tue Mar 02 06:02:23 1993 MODEL = Analect RFX – 40
SCNS = 16/16 GAIN = 8. 00 / 8. 00 RESD = 4. 000 APOD = NB - M DET = TGSC 0



FN : D TITLE : METHAMPHETAMINE HCl
DS1 : NEAT SAMPLE
DS2 : THIN FILM ON KBR PLATE
Y =WN Y=%T DATE = Tue Mar 02 06:30:33 1993 MODEL = Analect RFX – 40

Figure 5.3 Methamphetamine HI/HCl comparison.

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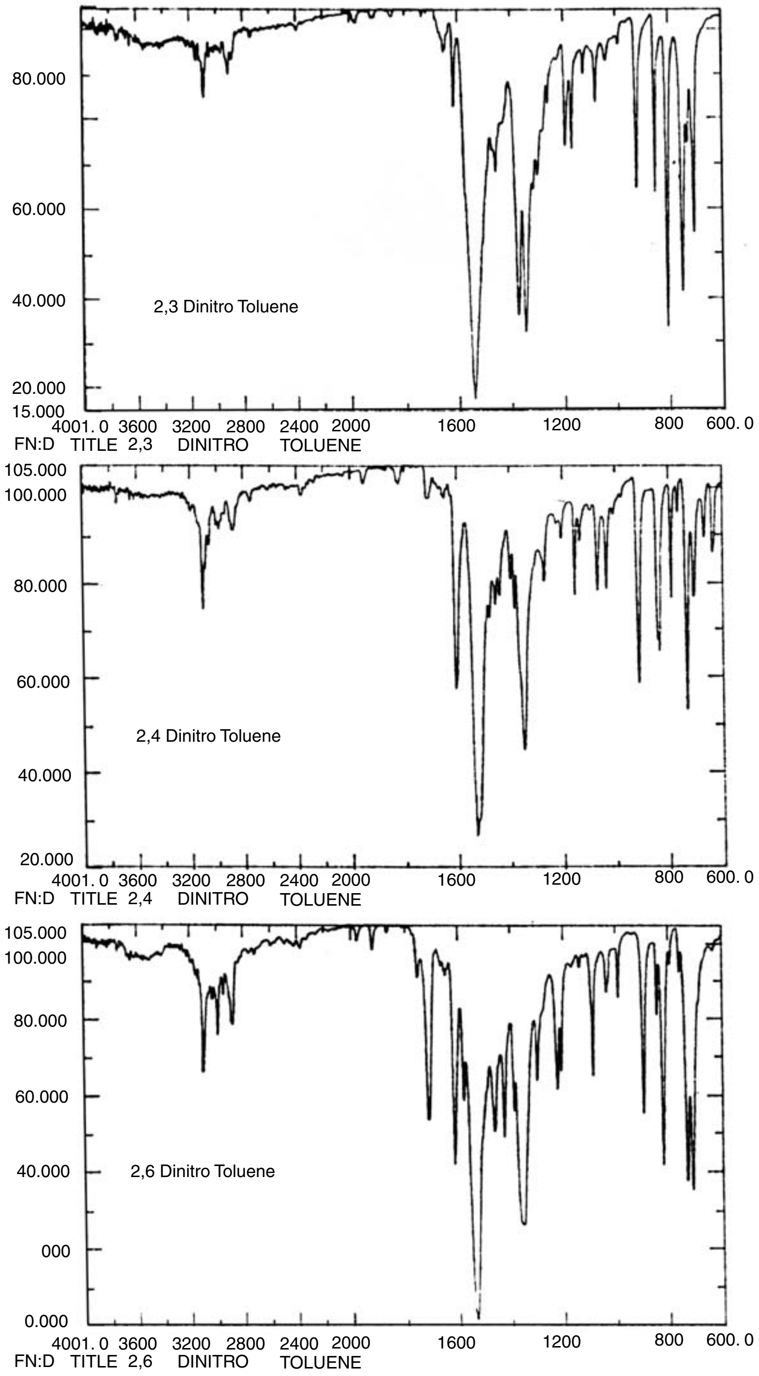

Figure 5.4 Dinitrotoluene isomer comparison.

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allows the reviewer to evaluate whether instrumental results are consistent with analytical conditions. If necessary, an independent examiner should be able to achieve the same results under the same test conditions. All data should contain, at a minimum, the examiner's initials, case number, exhibit number, solvent information, and date of analysis. The examiner should have the instrument print this information on the spectra at the time of analysis, if the instrument has the capacity to do so. For GC analysis, the calculated *RRt* value should be on the chromatogram or on the printout of the peak retention times. The divisions of the mass value axes on MS data should be such that the examiner can easily determine the mass value of each of the ions of the spectra. The wave number of the significant peaks of an IR spectrum should be labeled or should be easily determined by a peer reviewer. The examiner should have the instrument print this information at the time of analysis, if the instrument has the capacity to do so.

5.2.5 Analytical Schemes

The analytical schemes used to examine clandestine lab samples can be divided into solid and liquid schemes. Solids are usually precursors, reagents, or controlled substances and should be treated as unknown controlled substances. Liquid samples can be organic, aqueous, or a mixture of the two. They can be pure chemicals, reaction mixtures, or waste products and should be treated as if they contain a controlled substance.

It is common practice for operators to remove the labels from chemical containers and repackage chemicals into different containers. They place waste or finished product into empty chemical containers. Therefore, a chemist may find a container's label little more than insight into what chemical the operator possessed at one time. A container labeled ethyl ether may just as well contain a brown liquid with a chlorinated solvent odor, as the clear volatile liquid it is supposed to be. The chemist will need to modify his analytical scheme to identify the unknown mixture in this situation, as opposed to confirming the identity of a chemical from a labeled container.

All samples should be screened for the presence of controlled substances. The screening method used is up to the chemist and the capability of his laboratory. The screening method should not only detect the presence of controlled substances but should also include techniques that would tentatively or positively identify the presence of precursor chemicals, reagent chemicals, or reaction by-products commonly used or encountered in the manufacture of the controlled substances. If a controlled substance is detected, its identity should be confirmed. If the substance appears to be a precursor, reagent, or solvent, its identity should be established to the degree of certainty dictated by the circumstances.

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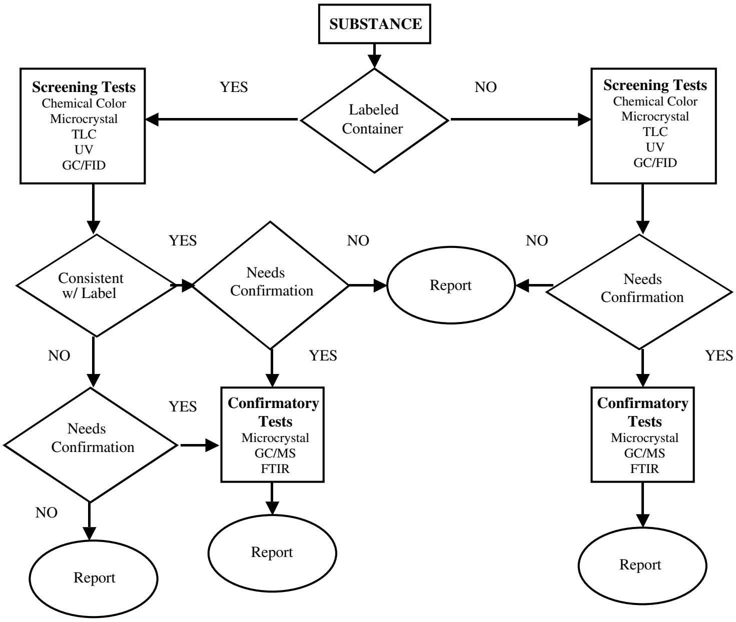

Figure 5.5 Unknown solid scheme.

5.2.5.1 Solid Samples

The analysis of solid samples follows the same analytical scheme as a controlled substance unknown. The same systematic analytical approach is used, as if the sample was an unknown and even if the identity of the substance is suspected. Sample analytical schemes should include screening for the presence of controlled substances, confirming the identity of controlled substances that have a high burden of proof, and confirming the identity of other substances to the extent their burden of proof dictates (Figure 5.5).

The sampling schemes for solid samples from labeled containers and loose solid materials are basically the same. Initially, the samples are screened using a combination of nonspecific tests. At this point, a decision is made. In the case of known samples, the following questions are asked: Is the sample consistent with the label? Does the sample contain a compound that needs a confirmatory examination? In the case of unknown samples: What does the sample contain? Does it need a confirmatory examination? The answers to these questions and the levels of specificity needed for particular identifications will guide the flow for the balance of the testing.

5.2.5.2 Liquid Mixtures

Liquid samples from clandestine labs deviate from the sample form normally encountered by the forensic chemist who deals with controlled substances.

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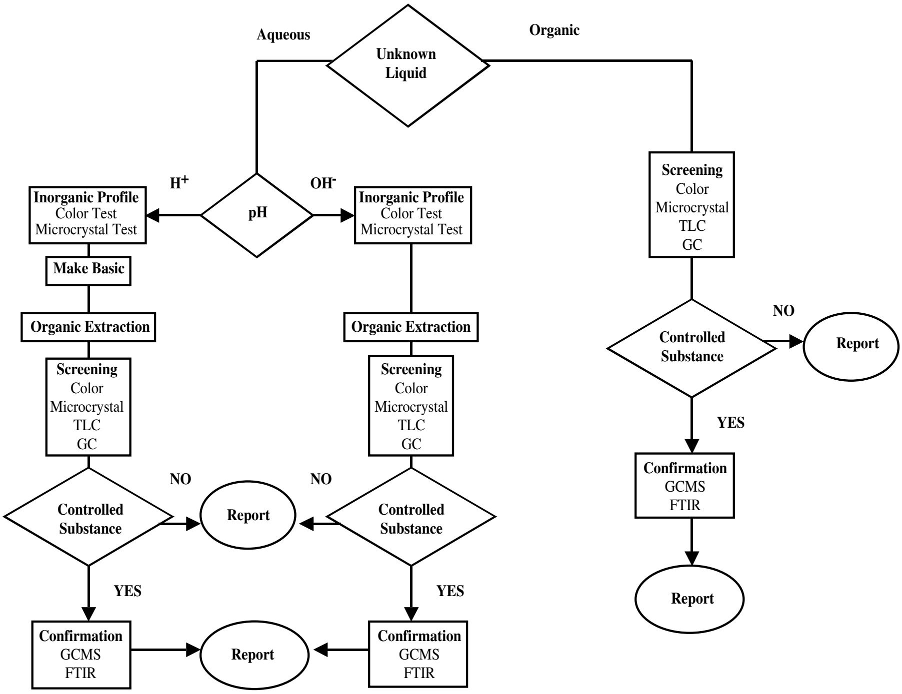

Figure 5.6 Unknown liquid scheme.

These samples, however, can give the chemist the most complete picture of the type of synthesis the operator was using. An in-depth analysis of these liquids can produce information about the product and by-products of the reaction as well as the precursor and reagent chemicals that were used in the synthesis. By evaluating the information that can be obtained from these liquid samples, the chemist can also determine at what step in the process the operation was at the time of seizure.

Liquid samples come in organic and aqueous forms (Figure 5.6). Both forms can be analyzed using the same analytic tools that were utilized for solid sample analysis. Organic samples are usually extraction solvents that may or may not contain a controlled substance. They are treated simply as a controlled substance exhibit in a liquid substrate. The analysis of unknown aqueous liquids requires a combination of organic and inorganic analytical techniques.

5.2.5.2.1 Organic Liquids. Organic liquids from clandestine labs can be reaction mixtures; extraction solvents (ether, Freon, chloroform, or petroleum products) that contain the final product; extraction solvents from which the final product has been removed, leaving only trace quantities of the final product; wash solvents that contain reaction by-products with traces

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of the final product; clean unused solvents, reagents, and precursors; or the final product.

The only way for the chemist to definitively say a clear liquid contains more than a single component is to analyze its contents. Just because a liquid looks, smells, feels, and tastes like Freon, does not mean there is not something dissolved in it. In almost every case, if the final product came in contact with the organic liquid, there will be a detectable amount of that substance in the liquid. It is the chemist's challenge to find it, if it is there.

The analysis scheme of organic liquids mirrors that of organic solid samples. Using chromatographic techniques, the sample is screened to establish whether or not it contains a controlled substance. A profile of the sample's contents is used to establish the liquid's place in the manufacturing process. Confirmatory tests are performed on compounds for which burden of proof is required. Finally, a report is generated.

5.2.5.2.2 Aqueous Liquids. Aqueous liquids can be reaction mixtures or waste products. Each type of liquid contains a wealth of information concerning the manufacturing process and will require organic and inorganic examinations. The inorganic profile of the liquid will guide the analysis.

Determining the pH of an aqueous liquid is the initial analytical step that provides the analytical chemist information concerning the liquid and the compounds that it may contain. As a general rule, acidic liquids are reaction mixtures, and basic liquids are waste material. Each type of liquid will have characteristic organic and inorganic compositions.

Once the pH of the liquid is established, the chemist must determine what type of information he desires from the sample. He should ask himself: Does he simply want to isolate and identify any controlled substances from the sample? Does he want to extract all of the organic constituents from the solution and try to establish a synthesis route? Does he want to identify the inorganic components of the aqueous solution? Or, does he want to forego analyzing the sample, because it is similar to 12 other samples from the same location? The answers to these questions will determine the analytical sequence as well as any extraction techniques that may be used.

Establishing the inorganic profile of an aqueous sample is the next segment of the analysis. This can be accomplished by using the same methods described in the inorganic analysis section. The chemist is looking for the type of acid or base that was used in the reaction as well as any inorganic reagents that may indicate a particular reaction route. A series of chemical color tests and microcrystal examinations can provide the chemist with a sense of what the sample contains and where it fits into the manufacturing process. Three drops of sample may be all that is necessary to provide the chemist with a complete inorganic profile of the aqueous liquid.

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As a general rule, the chemist should screen all aqueous liquids for their organic component content. Acidic liquids are generally reaction mixtures, and potentially, they can contain large quantities of the controlled substance. Basic liquids are generally waste products that will contain reaction by-products characteristic of the manufacturing process used as well as a detectable amount of the controlled substance that was manufactured. The fact that there were no organic components detected in the sample is significant information.

Most controlled substances and precursors are soluble in acidic aqueous liquids and may be visually undetectable. Thus, the analytic chemist should expect high concentrations of these chemicals in acidic solutions. To remove the controlled substance from the aqueous solution, he should change the pH of the solution and extract it with an organic solvent. This will remove the basic and neutral organic compounds from the aqueous solution. The organic extract can then be analyzed as if it were an unknown organic liquid.

There may be instances in which the acidic and neutral compounds that the aqueous liquid may contain will be of significance. In those situations, the acidic liquid should be extracted with an organic solvent prior to making the aqueous solution basic to extract any controlled substances that may be there. Again, the organic extract is analyzed as if it were an unknown organic liquid.

The analysis of the organic extracts of aqueous samples mirrors that of organic liquid unknowns. Chromatographic techniques are used to screen the sample to establish if the sample contains a controlled substance. The chromatographic profile of the sample's contents is used to establish the liquid's place in the manufacturing process. Confirmatory tests are performed on samples that contain compounds for which burden of proof is required. Finally, a report is generated.

5.2.5.3 Chromatographic Screening

Chromatographic techniques include gas and liquid chromatography. They are useful tools for analyzing organic liquid samples. These techniques allow the chemist to obtain a profile of the organic makeup of the unknown with a single test. The chemist can then determine whether a liquid contains one component, a mixture of a dozen, or none. Using retention time data, the chemicals and reaction by-products associated with various clandestine manufacturing methods can be identified. The peak areas from the chromatograms can be used to establish concentration ratios leading to quantitative estimates.

With chromatographic analysis, the chemist can quickly establish the number and probable identity of the components in an organic mixture. Under general screening parameters, the unknown's solvent will elute from the chromatography column with the solvent the chemist uses to prepare his sample for analysis. If the analyte is a clean solvent, the resulting chromatogram will appear to be blank. The identity of the analyte solvent can be

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determined by modifying the chromatograph's acquisition parameters to enable separation of the low-boiling-point solvents.

The significance of the peaks in the chromatogram is determined by the analytical chemist's interpretation of data. The relative amounts of the compounds will depend on the sample preparation technique used. The chemist should use an established sample preparation scheme so that the results from all of the samples of a case can be compared. For example, a sample of an extraction solvent will contain a large amount of final product, possibly overloading the chromatographic column. If the sample is a waste solvent, there will be only trace amounts of product present. If the chromatographic peaks of samples that were prepared identically produce different peak areas for a peak that elutes at the same time as methamphetamine (e.g., Sample 1 area is 500 counts; Sample 2 area is 500,000 counts), the analytical chemist could infer that Sample 1 was a waste solvent, and Sample 2 was an extraction solvent containing the product.

The symmetry of a chromatographic peak can provide information concerning the sample. In some instances, it can be used as a presumptive test to establish whether the compound in a solution is the freebase or is in a salt form. Generally, the peaks of freebase and neutral compounds produce sharp symmetrical GC peaks when using nonpolar columns commonly used in drug and ignitable liquid analyses. Sulfate salts and HCl salts of low-molecular-weight compounds chromatograph poorly, producing asymmetrical peaks that can tail badly. High-molecular-weight salt compounds do not demonstrate this tendency.

If there is an indication of a controlled substance, its identity must be confirmed by a documentable technique. The type of information desired will determine the confirmatory route taken. If only the identity of the compound is desired, purification extraction prior to confirmation may be desirable. If the identity of all of the components of the mixture were desired, a simple dilution and analysis would be appropriate.

The chromatograms from similarly prepared samples can be used to establish a common origin or manufacturing technique. The pattern and ratio of product, precursor, and by-product peaks can be used to determine common origins of samples or positions in the reaction sequence. Samples from the same case can be compared in a manner similar to the way a chemist compares ignitable liquids and the residues extracted from fire debris.

5.2.6 Extractions

Extractions are used to separate the compound of interest from the rest of the sample. The type of extraction scheme used will depend upon the compound of interest, the sample matrix, and the information desired from the resulting analysis. In some cases, multiple extraction techniques are necessary

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for separating the substance of interest from the remainder of the sample. In other instances, instrumental analysis is the only way to separate compounds with similar chemical properties for confirmation.

In devising an extraction scheme, the chemist must decide what he wants to isolate and the form it will be in after it is separated. The answers to these questions are in the statutes under which the chemist is working. Some statutes require only the presence of the controlled substance without regard to its salt form, isomer status, or purity. Other statues are specific when it comes to identifying a controlled substance by its salt form, structure, isomer form, or purity. In these cases, be sure that extraction does not alter the form.

The basic types of extractions include physical extractions, dry washes, dry extractions, and liquid/liquid extractions. In this section, the generic applications of the different types of extraction will be described. Specific extraction procedures are described in Appendix L.

5.2.6.1 Physical Extraction

Physical extractions are the simplest. They involve physically removing the substance of interest from the balance of the sample. The isolated substance is then analyzed by the technique the examiner deems appropriate.

Physical extraction is appropriate when the examiner observes particles of different sizes, shades, and consistencies within the sample. The particles are physically or manually separated from the bulk sample by using stereomicroscopes, tweezers, sieves, or other devices designed to physically isolate particles of different sizes.

5.2.6.2 Dry Wash/Extraction

Dry washes and dry extractions are different versions of the same process. The only difference is the substance that is removed from the sample matrix. With a dry wash, a solvent is used to dissolve and remove adulterants and diluents from the sample matrix, leaving the compound of interest. With a dry extraction, a solvent is used to dissolve and remove the compound of interest from the sample matrix.

5.2.6.3 Liquid/Liquid Extractions

The ability of a substance to dissolve in a liquid can change with the liquid environment. Liquid extractions utilize these solubility characteristics to separate a substance from a mixture. Listed in Appendix L are the general solubility rules used for liquid/liquid extractions.

During a liquid/liquid extraction, the sample is initially mixed into an aqueous solution. The aqueous liquid is washed with an organic solvent in which the compound of interest is not soluble but the diluents and adulterants are soluble. The organic liquid is separated, and the pH of the water is

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changed in such a way that the compound of interest is made insoluble in the water solution. An organic solvent is used to separate the substance from the water.

Care must be taken when selecting the acidic environment and the organic solvent used in liquid/liquid extractions. Some drugs are subject to ion pairing. This means that the hydrochloride salt form of the drug is soluble in chlorinated solvents (i.e., chloroform) and will choose the chlorinated solvent over an acidic environment with a high chloride concentration (i.e., HCl).

Ion pairing can be used to the examiner's advantage when there are multiple basic drugs within a matrix that need to be isolated. If one of those drugs is subject to ion pairing, it can be isolated from the other drugs that, under normal circumstances, could not be separated.

In some instances, the compound of interest cannot be isolated, because the sample matrix contains multiple drugs of the same salt type. In these instances, a combination of techniques may be necessary to isolate the component of interest. An example of a combination extraction would be performing a TLC separation of the final extract of a liquid/liquid extraction. The silica gel around the spot corresponding to the compound of interest is physically removed from the TLC plate. A dry extraction or another liquid/liquid extraction is performed to isolate the substance from the silica gel.

5.2.7 Isomer Determination

Once the identity of a substance has been confirmed, the analysis is usually complete. Most statutes are written to include isomers, salts, and salts of isomers when defining a controlled substance. However, there are instances when the statute is specific in defining the controlled substance. They specifically define the structural configuration or the optical isomer of the compound that is controlled. In these instances, additional work may be necessary to satisfy the statutory definitions.

"Isomer" is a generic term that can encompass a number of different meanings. Isomers are compounds that have the same molecular formula but a different structural formula. The differences can be obvious, as in the case of structural isomers, or subtle, as with stereoisomers.

Structural isomers are compounds that have the same molecular formula but a different structural formula. Examples of structural isomers are ethyl ether and ethanol. Each compound has the molecular formula of C_2H_6O. However, the structural formulas are CH_3OCH_3 and CH_3CH_2OH, respectively. Their different structures give them different chemical and physical properties that allow them to be differentiated through various instrumental techniques.

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Geometric isomers are isomers that result from the positioning of two different functional groups attached to different ends of a double bond. The double bond prevents rotation, creating a *cis* (functional groups on the same side of the double bond) and *trans* (functional groups on opposite sides of the double bond) configuration. Geometric isomers have similar chemical properties but different physical properties. GC and IR can be used to differentiate them through analysis.

Optical isomers are compounds that have the same structural formula. The only difference is the arrangement of functional groups around a chiral (asymmetric) carbon. This difference affects the rotation of plane polarized light. One configuration will rotate light to the right (*d*, dextrorotatory) and the other to the left (*l*, levorotatory). Otherwise, the chemical and physical properties of these isomers are identical. Microcrystalline tests and instrumental analyses of the derivatized compound are two methods available to forensic labs to use to differentiate optical isomers.

5.2.7.1 Microcrystal Examination

Microcrystal examinations used to determine the orientation of optical isomers are rapid analytical methods that require only a microscope and the necessary reagent chemicals. The compound in question reacts with an inorganic reagent chemical to form a complex that is insoluble in the test solution. The resulting complex has a characteristic crystal shape that can be observed under the microscope. Racemic mixtures (mixtures that contain both optical isomers) produce different microcrystals than single isomer compounds. The microcrystals of a single optical isomer generally cannot be distinguished from the microcrystals of the other optical isomer.

If the chemist needs to know the optical orientation of a compound, he can perform a mixed crystal test if the microcrystals of a single optical isomer were observed. A mixed crystal test involves placing an equal amount of a compound with a known optical orientation with the unknown sample (e.g., a small amount of known *d*-methamphetamine is combined with the same amount of unknown single isomer methamphetamine). The microcrystal test is performed on the known/unknown mixture. If the resulting crystals are single isomer crystals, the unknown has the same optical orientation as the known. If the resulting crystals are the crystals obtained from a racemic mixture, the unknown is of the opposite optical orientation.

5.2.7.2 Derivatization

The other method of determining the optical orientation of a compound is through derivatization. In this technique, the derivatizing reagent reacts with the compound at a reactive on the molecule, usually at a nitrogen site or at a hydroxyl group. The addition of the derivatization agent to the molecular

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structure of the compound alters the chromatographic properties of the compound to such an extent that optical and stereoisomers can now be chromatographically separated.

Derivatization not only alters the chromatographic properties of the derivatized compound, but also alters the resulting ion patterns of the mass spectra between the derivatized isomers, making them differentiable. Shown in Table 5.5 are the eight most prominent peaks of the *n*-trifluoroacetyl-(S)-prolyl chloride (TFAP) derivative of the amines commonly encountered at clandestine drug labs or in the controlled substance samples. Not only are the mass spectra differentiable, but also, each is distinguishable chromatographically.

5.3 Quantitation

Once the identity of the controlled substance has been established, it may become necessary to determine the exact amount of that substance that is the sample. This may be necessary for a number of reasons. The governing statutes may require that the exact amount of controlled substance be determined. The percentage of the sample that is a controlled substance may influence the chemist's opinion as to whether the substance is finished product, waste material, or something in between. Or, the chemist might just want to know.

As a general rule, there is no statutory requirement to perform a quantitative examination on controlled substance samples. Quantitation is used as an investigative tool or is done as part of a laboratory's internal security policy. With a few exceptions, criminal statutes regulate only the possession of a given substance. The concentration of a sample does not affect guilt or innocence.

The concentration of a sample may become an issue during the sentencing phase of a trial. Some statutes provide enhanced penalties for possession of a substance over a given quantity. The words "possession of *X* grams of compound *Y*" are distinctly different than "*X* grams of substance containing compound *Y*." This wording may affect whether a quantitative exam is required to establish a sentence of 1 year or 10.

There are numerous quantitative techniques that the chemist can use to determine the concentration of a substance in a sample. Before the chemist can begin his quantitative analysis, he must determine the type of information he is trying to obtain. Does he want to accurately know how much controlled substance is in a given sample? Or, does he want his analysis to reflect the amount of substance the operator could obtain from the sample? The answer to these questions will determine the type of quantitation method used.

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Table 5.5 TFAP Derivative MS Table*

Isomer	PK1	PK2	PK3	PK4	PK5	PK6	PK7	PK8	Mol. Ion
l-Ephedrine	58	166	251	252	69	42	167	41	None
d-Ephedrine	58	166	252	251	69	42	167	77	None
l-Pseudoephedrine	58	166	251	252	42	77	167	43	None
d-Pseudoephedrine	58	166	252	251	69	42	77	41	None
(1) Propylhexadrine	166	58	69	125	55	41	182	167	348
(2) Propylhexadrine	166	58	69	41	125	55	182	167	348
l-Amphetamine	166	237	194	91	118	69	44	167	None
d-Amphetamine	166	237	194	91	118	44	69	41	None
l-Methamphetamine	58	166	251	91	42	41	69	96	None
d-Methamphetamine	166	58	251	91	69	41	42	119	None
(1) *p*-Methoxyamphetamine	148	166	194	121	44	69	41	167	358
(2) *p*-Methoxyamphetamine	148	166	121	194	149	44	69	41	358
(1) 3,4,5-Trimethoxyamphetamine	208	166	418	194	181	193	209	167	418
(2) 3,4,5-Trimethoxyamphetamine	208	166	418	194	181	193	209	167	418
(1) 2,4,6-Trimethoxyamphetamine	181	208	166	182	44	121	209	69	418
(2) 2,4,6-Trimethoxyamphetamine	181	208	166	182	209	120	69	44	418
(1) 4 Bromo 2,5 dimethoxtamphetamine	166	285	256	194	44	237	468	69	467
(2) 4 Bromo 2,5 dimethoxtamphetamine	166	285	256	194	44	237	69	468	467
(1) 4 Methyl 2,5 dimethoxtamphetamine	192	166	402	194	193	165	69	44	402
(2) 4 Methyl 2,5 dimethoxtamphetamine	192	166	402	194	193	165	69	44	402
(1) Methylenedioxyamphetamine	162	166	194	135	372	163	69	44	372
(2) Methylenedioxyamphetamine	162	166	194	135	44	77	69	163	372
(1) Methylenedioxymethamphetamine	166	58	162	251	163	69	135	77	386
(2) Methylenedioxymethamphetamine	58	166	162	69	163	135	251	96	386

Source: From McKibben, T., *J. Clandestine Lab. Investigating Chemists Assoc.*, 2, 1, 13, January, 1992. With permission.

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The four basic methods of quantitating the amount of controlled substance in a sample are microscopic examination, gravimetric comparison, UV analysis, and GC analysis. Below are the generic descriptions of the various quantitation methods.

5.3.1 Microscopic Examination

The quickest and most subjective solid sample quantitative method is accomplished through microscopic examination. In this technique, a sample is placed on a microscope slide and diluted with a solvent to which the components are insoluble. The examiner estimates the percentages of crystals of the various substances in the sample under observation. This is the most subjective, least precise, and least accurate method. It is subject to the examiner's ability to recognize the microscopic crystalline form of the controlled substance under consideration. The uniformity of the bulk sample also affects the accuracy and reproducibility of the results.

5.3.2 Gravimetric Techniques

Gravimetric analysis provides a rapid means with which to determine the approximate amount of controlled substance in a sample. This technique can be used on organic and aqueous samples. This technique also mimics the method operators use to extract the final product from reaction mixtures or extraction solvent. Therefore, it provides a practical approximation of how much of the final product the operator could expect to recover from the sample.

Gravimetric techniques can be performed in conjunction with the extraction phase of an analysis. The examiner weighs or measures the volume of the sample to be extracted prior to the extraction process. He obtains a weight of the extracted substance prior to any confirmatory tests being performed. The ratio of the postextraction weight to the preextraction weight provides the percentage of the item that is the controlled substance.

A limiting factor to the precision and accuracy of this technique is the efficiency of the extraction solvents. If they do not effectively remove the diluents and adulterants, the calculated controlled substance percentage will be high. If the solvents do not efficiently and completely isolate the controlled substance, the percentage will be low.

An advantage of gravimetric techniques is that the identity and composition of the final extract can be confirmed. If all the diluents and adulterants have been removed from the matrix, the resulting residue can be analyzed for purity and then identified.

The examiner must be aware of the salt form the controlled substance is in before and after the extraction process. This will affect the percentage

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calculated, because the molecular weights of the salt form differ from the molecular weights of the freebase. For example, a 100% pure sample of cocaine hydrochloride contains 89.38% by weight freebase cocaine. The examiner must take into account the mass of the salt when calculating the percentage of controlled substance in the sample or must qualify the conclusion by stating the salt form of the substance identified.

5.3.3 UV Techniques

The use of UV light provides an effective method to quantitate a sample, if it has a single UV absorber. If the sample has components with overlapping UV absorbances, the instrument cannot determine which compound is contributing to the absorbance. Compounds also absorb UV radiation at different rates. Therefore, UV methods are not conducive to quantitating mixtures.

Simple "yes" and "no" concentration comparisons can be accomplished with the use of UV techniques. These comparisons are conducted in association with a product tampering case, in which the product in question may have been diluted or altered. Comparison of the UV spectra of the item in question to a known reference sample can indicate if the unknown has been diluted or altered. The compositions of both samples should also be confirmed through separate examinations.

A detailed examination can determine the concentration of the substance in question. To accomplish this, the examiner obtains the UV spectra for a series of solutions with a known concentration of the substance in question. The absorbance values are placed on a concentration versus absorbance graph. A solution of the unknown is prepared and analyzed. The absorbance value is placed on the graph to determine the concentration of the substance in the solution. This value is then used to calculate the percentage of substance in the unknown.

UV techniques done properly are precise. However, the accuracy of the results for multicomponent mixtures may be in question because of the interference of the UV absorbance of other compounds in the sample.

5.3.4 GC Technique

The use of GC for quantitation provides the most accurate and precise results compared to the other analytical techniques discussed. This technique provides the examiner the ability to isolate and quantitate a specific compound in a single method. The identity of the chromatographic peak can be confirmed at the time of the analysis or by analyzing the test solution with a GCMS. The same chromatographic conditions should be used during the confirmation test so that a direct correlation between the two techniques can be made.

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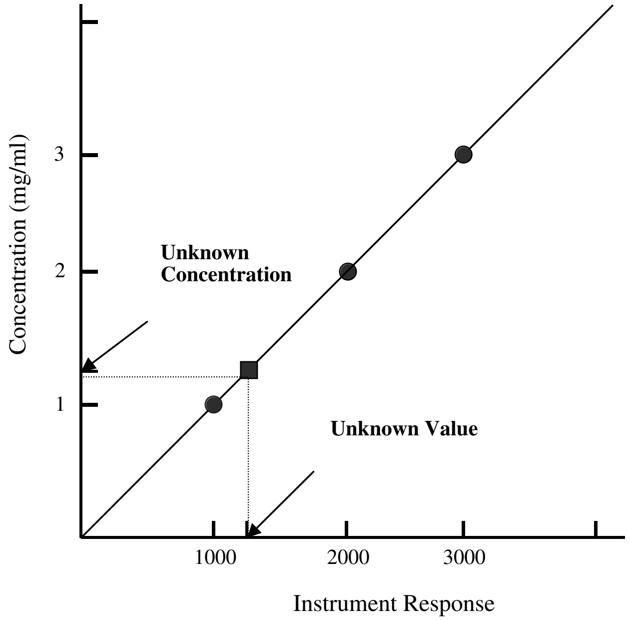

Figure 5.7 Beer's law plot.

Traditionally GC quantitation uses a concentration versus peak area plot to establish the concentration of an unknown solution (Figure 5.7). If the peak areas of the serial dilutions of a substance are charted, the concentration of an unknown solution can be determined from its instrumental responses. This method uses the relationship between the concentration of a sample and the instrumental data to calculate the concentration (i.e., doubling the sample concentration will double the GC peak area). A series of diluted samples is prepared and analyzed on the GC. The resulting peak areas are plotted on an *X*/*Y* graph with their corresponding solution concentrations. The unknown solution is analyzed in the same manner. Its concentration is obtained by using the graph generated by the known solutions.

The increase in the precision and accuracy of modern instrumentation has allowed the analytical chemist to reduce the number of reference samples necessary for GC quantitation. The relative response GC method of determining sample concentration uses the ratio of the compound's and internal standard's peak areas, known sample concentrations, and algebra. Two GC injections using this method can provide the same results as multiple injections using the serial dilution method. This procedure is based on the predictable relationship between sample concentration and the peak area of a chromatogram, i.e., doubling the sample concentration will double the resulting peak area of the chromatogram.

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The use of the area concentration ratio to determine the concentration of a solution is dependent on the precision of the volumes injected into the GC for analysis. Small deviations in injection volumes will affect the accuracy and precision of the analysis.

To compensate for any deviations in injection volumes that may occur, a known concentration of internal standard is placed into the standard and unknown solutions prior to analysis. The concept of a given concentration producing a given peak area is just as true for the internal standard as the samples in which they are placed. This being the case, the ratio of peak area of sample to peak area's internal standard (IS) will not change for a solution, no matter what volume is injected into the GC.

When quantitatively analyzing organic liquids, the chemist must dilute the sample until the unknown sample produces approximately the same compound-to-internal-standard ratio that exists in the standard solution. With the known dilution factor, the chemist can calculate the original concentration. If the sample in the previous example had a 20:1 dilution factor, the concentration of the original sample would have been 25.2 mg/ml.

By converting the concentration term into its basic units of weight (W) and volume (V), the concentration equation can be manipulated into an equation that describes the percentage of the unknown that contains the target compound.

5.4 Summary

The complete analysis of clandestine lab samples is an essential portion of the forensic investigation. The information derived from the testing can only be obtained through the scientific examination of the evidence. This information is used to meet the burden of proof required to establish the presence of a controlled substance, beyond a reasonable doubt. The cumulative effect of the examinations that can only establish a preponderance of evidence can be used to formulate expert opinions concerning the operation.

The tools the clandestine lab chemist uses to analyze these samples are the same tools that the forensic chemist uses to analyze controlled substance samples. The only difference is the clandestine lab chemist may apply certain techniques in a method to produce more detailed information concerning the sample. The scope of the analysis goes beyond the forensic chemist's desire to determine whether or not a controlled substance exists. The clandestine lab chemist needs to know what else is in the sample so that he can develop opinions concerning the operation.

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Opinions

6

A clandestine lab is a Pandora's Box of illegal activities. Controlled substances are produced using household chemicals mixed with ordinary utensils in what some have called a "kitchen of death." What appears at first glance to be simply atrocious housekeeping or even just a hobby gone awry may actually be the final step in the production of many of the drugs sold on the street or the explosives used in various forms of domestic terrorism.

Everyone has his own image of what a clandestine lab would look like. The man on the street, from which a typical pool of jurors is drawn, will more than likely report images of smoking and boiling chemical reactions using scientific equipment in a hidden laboratory, such as Frankenstein's birthplace. As has been demonstrated in the previous chapters, this is far from the case. An expert is called to understand what he sees at the crime scene and to draw a mental picture for others as well. Remember that all experts are, first, investigators, but not all investigators are able to become experts.

A forensic expert must first assemble enough pieces of physical evidence to demonstrate that a clandestine lab exists. He must be able to combine the known facts to present a scenario of the "What? Where? Why? and How?" of the operation. His knowledge base is broad enough to acknowledge that other explanations could exist for the combination of chemicals and equipment found at a location. He considers the totality of circumstances of the case and concludes with a schematic of the most likely arena in which the manufacture of a controlled substance took place.

Forensic evidence and the opinions generated by it are used to supplement or answer the basic *Who? What? Where? When? Why?* and *How?* questions involved in any criminal investigation. While able to address objective questions, there are still limits on subjective ones. The expert's job is to use the forensic evidence to compile, evaluate, and render an opinion concerning the facts of each case as presented. The expert may be involved in the case

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from the beginning, or he may be brought in at the end to evaluate other expert opinions. Either way, the process is basically the same.

In Chapters 4 and 5, the forensic chemist who was trained in clandestine lab issues was presented as the ultimate expert. He has a significant knowledge base to draw from in presenting expert testimony and generating opinions. However, he is not the only source of expert opinions. Criminal investigators, bomb technicians, and hazardous materials specialists who regularly deal with clandestine lab issues can generate qualified opinions. While such individuals may not know the specific theories concerning the chemistry involved, their experience and training provide the knowledge base that a given set of chemicals and equipment can be used to produce a controlled substance.

In this chapter, the opinions generated by experts that are used during arrest and prosecution of clandestine labs are addressed. From the forensic evidence collected, experts are able to study a clandestine lab and afford opinions to the Court. Further addressed in this chapter are the evaluation and the opinion-making processes, and hopefully, insight will be provided into them. Sources of information that are needed to formulate an opinion will be listed. Questions the forensic expert needs to derive from that information will be addressed. These questions most significantly include the following:

What is the operator making?
How is the operator making it?
How much (quantity) could the operation produce?

6.1 The Questions

Clandestine lab experts are forensic investigators who come from a variety of functions within the criminal justice community. They may be peace officers or forensic chemists. They may or may not carry guns and have arrest authority. Whatever their functions, their missions are basically the same — collect and objectively evaluate information concerning a clandestine lab operation.

The expert must deal with the same *Who? What? Where? When? Why?* and *How?* questions as the investigators. Experts simply approach the question from different perspectives. Combining information concerning physical evidence and scientific principles to objectively evaluate the operation is their forte, and they use this straightforward angle of the case to formulate and provide their opinions. Some questions can be answered at the scene; others can only be addressed and determinations made after the laboratory examination is complete. The expert uses this combination of answers to all

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of the questions to form his ultimate opinion concerning the operation as a whole.

The opinion process begins with the affidavit for a search warrant. Using the information provided by the lead investigator, the expert renders an opinion as to whether there is sufficient information with which to establish the existence of a clandestine lab. The expert's opinion is a key factor in this part of the legal process, and it is used to support the investigator's conclusion of the facts. If flawed, the opinions presented could later be raised in court by defense counsel as "motions to suppress evidence" due to lack of probable cause. It is, therefore, necessary to exhibit extreme care when determining that certain conditions exist and that they point irrevocably in the direction of a clandestine lab operation.

Experts talking to investigators about what (on the surface) seems to be a general opinion about clandestine labs would be well advised to document these conversations. The expert's name may be placed into an affidavit without his knowledge. The statements attributed to him may be totally wrong, misinterpreted, or even never uttered in the first place. His supporting documentation of what was said during the preliminary conversations may become necessary if the expert is asked about statements he made in the search warrant affidavit. Obviously, this could crucially affect his future credibility.

The search of the suspected clandestine lab site is the pinnacle of the investigation. Emotions run high, people may be stressed and tired, and answers are demanded. Before an expert gets out of his car, investigators want to know: *What are they making in there? How are they making it? How much product is there? How much could they make in a...?* Unfortunately, many novice clandestine lab investigators really expect answers to these questions immediately.

In these instances, the expert does not have sufficient direct knowledge of the situation to render an opinion. At best, he should only make qualified generic statements concerning what could be made and possibly how it was being made, using the limited information he has. The chaos of a clandestine lab scene does not provide an atmosphere in which to render any type of objective opinion. Opinions concerning the specific details of the operation should be rendered only after the physical evidence can be evaluated in an objective manner, which is after the fact only.

Many types of opinions can only be generated from the laboratory analysis of evidentiary samples. Some are a result of generalities that do not require the support of analytical data. For example, just because a red powder is found at the scene of a suspected ephedrine reduction lab does not make the powder the critical red phosphorus. It is essential that if an analytical chemist is going to render an opinion concerning a clandestine lab, he must have the analytical data to support it.

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The forensic expert must remember the laboratory analysis, and his opinions should be able to withstand peer review. A component chemist or other forensic expert should be able to review the facts of the case or the laboratory data and draw the same conclusion as that of the original expert. Alternative opinions can and do exist, as is evidenced by prosecution and defense differences. But the information must support the opinion, or the opinion is worthless.

6.1.1 Who?

One of the initial questions in any investigation is: *Who was involved?* This simple investigative question can be answered to one degree or another by looking at the people detained or living at the scene. Placing them at the scene is one thing but connecting them to the lab operation may take a forensic expert. Latent prints can be used to establish who had access to the lab equipment and chemicals. In some instances, laboratory analysis of a suspect's clothing can be done to detect drug and chemical residues, which can be used to connect him to a lab operation. The analysis of the handwriting on paperwork associated with the operation can also be used to establish a link between the operation and people who were nowhere near it at the time of seizure.

6.1.2 What?

The most common what question is, *What are they making?* Asking the operator is the easiest way to obtain this information. However, operators have been known to be less than truthful, uncooperative, or simply unaware of the identity of the final product. That is where the forensic expert comes into play. He combines the information from the paperwork, available chemicals and equipment, as well as the laboratory analysis of items associated with the operation to provide an objective opinion concerning what was being produced.

Some lab operations perform only a portion of the process. This is done to avoid compromising the entire operation. In these instances, the *What step in the process are they?* question may play a significant role in the investigation. The expert utilizes the same information to determine what was being made and then rolls that information into an opinion concerning in what stage of the process was the operation.

6.1.3 When?

When were they cooking? This is not a question that is conducive to traditional forensic techniques. Other than being caught in the act, the forensic expert

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cannot provide much insight. Traditional investigative techniques provide the best methods of answering this question.

6.1.4 Where?

Where was the lab? The location of all or any given segment of the operation can be determined through forensic investigation. The position at the scene of the lab, the chemicals, or where the equipment was located can easily be documented. Forensic investigation and analysis can demonstrate where each portion of the process was conducted as well as where the suspect was putting his waste products. Even if the lab was dismantled and removed from a location, analyses of the residues left on floors, or even stains left on the walls and counters, can be used to determine where the lab was located and what was being produced. Shown in Figure 6.1 is an example of how the location of a makeshift ventilation system can be used to demonstrate the location manufacturing operation.

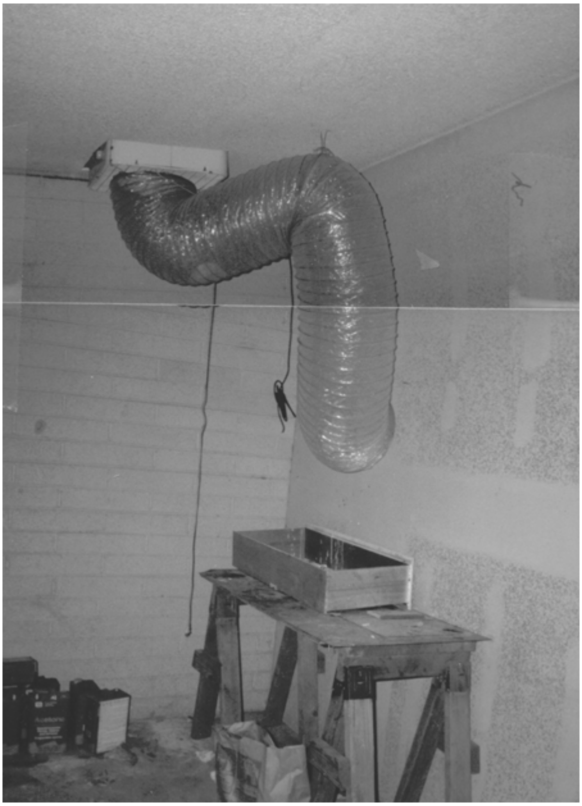

Figure 6.1 Makeshift ventilation.

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The questions, *Where was he getting his chemicals and equipment?* or *Where was he storing excess chemicals and equipment?* can be answered by reviewing documents seized from the scene or other locations associated with the operation. Operators are generally pack rats and save everything. Where you will find evidence concerning the operation is only limited to the imagination of the operator and the investigator searching the scene.

6.1.5 Why?

On the surface, *Why?* is a simple question to answer. Money, drugs, or both is the answer to the basic *Why are they manufacturing?* question. However, the forensic expert needs to dig a little deeper and ask a few more questions in this same vein: *Why did the operator use this method of manufacture? Why did he use a particular chemical supplier? Why did he use the equipment he used?* The answers to most of these *Why?* questions is subject to conjecture. However, experts, by their nature, have the latitude to speculate. They are expected to use their training and experience to evaluate the facts of the case and present an educated theory concerning the why's of the operation.

6.1.6 How?

Many of the "How" questions need a technical expert to answer. They all have the potential to be asked in court. The lead investigator may want on-the-spot answers to many of the same questions. Unfortunately, many can only be completely answered after laboratory analysis and data interpretation. The how questions include: *How were they making the product? How much product was there? How much could they make per batch or over a given period of time? How much could they make with the seized chemicals and equipment?*

6.2 Information

Information is key to answering any question. Answering questions without information is like putting a puzzle together without enough pieces. With a puzzle, the more pieces that exist, the closer to the complete picture that is created. The more information an expert knows about a clandestine operation, the closer he can come to painting a complete picture of the operation.

The clandestine lab expert has a needs triangle similar to that of the clandestine lab operator (Figure 6.2). The clandestine lab operator needs chemicals, equipment, and knowledge to make the operation work. The clandestine lab expert needs information from the scene, from the laboratory analysis, and from the knowledge gained from training and experience. Scene information provides knowledge concerning the operation in general. Labora-

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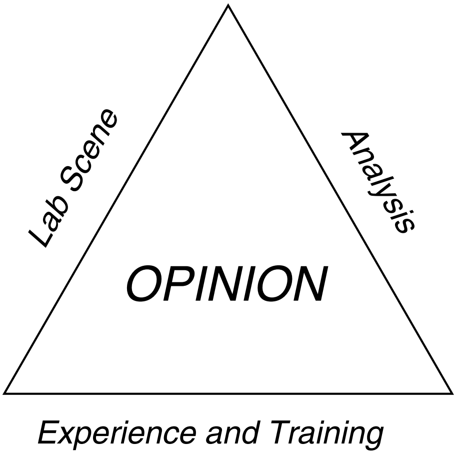

Figure 6.2 The opinion needs triangle.

tory analysis provides information concerning the specifics of any given sample. The expert's experience and training allows him to piece together all the information to complete the picture of the operation.

6.2.1 Scene Information

The information gathered at the scene of a clandestine lab has a number of functions. First, it corroborates the prior information used to establish probable cause in obtaining the search warrant. Second, it guides the direction of the on-scene investigation. Third, it helps the analytical chemist devise the analytical schemes he will use during the testing of the samples that are sent to the laboratory. Finally, it will be used as a basis for the expert's testimony concerning the workings of the operation.

The information gained during the initial scene walk-through sets the tone for the balance of the scene processing. The expert's initial impressions of what the operator was making and how he was making it help to determine what type of search will be conducted. Drug labs may take one approach. A cautious and different path may be necessary with suspected explosive labs. If no lab is initially apparent, a different search tactic is taken.

The common thread in all searches is that the observations and the physical evidence guide the on-scene investigation. The desire to put someone in jail should not be so great that the expert misinterprets or misrepresents the presence of common items to justify the presence of the police. Search warrants for the wrong location have been granted on poor information. This situation should never deteriorate to where the forensic expert is looking for whatever evidence it takes to allow the law enforcement agency to save face. On the other hand, even if the operation is not readily apparent, the

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forensic expert should be as creative in his search techniques as the operators are in hiding and disguising the labs.

The analytical chemist uses the information provided by the scene chemist or expert to devise his analytical schemes. He reviews this information to determine what samples require examination and what types of testing are appropriate. The analytical chemist also uses the scene information to estimate the amount of final product the operation could produce. This information can also explain reaction by-products that are not normally encountered in the reaction mixtures from that type of lab.

The opinions rendered from scene information are forensic evidence, and they must stand up to peer review. Documentation of the observations made at the scene is a critical component. Photographs, sketches, and inventory lists can be used to support the expert's opinions rendered in the report. An independent expert should be able to review the scene documentation and come to the same conclusion as the person generating the report. That is not to say that the independent expert may have a differing opinion, only that the documentation supports the report's conclusion when looked at by outside review.

The scene chemist must take care not to overstate his opinions concerning the scene. It is easy to get caught up in the frenzy of the moment and provide an opinion that is not completely supported by the facts. Producing a written report, after he has had time to objectively evaluate all of the information concerning the operation, is the wisest method of disseminating the opinions concerning the operation. The written report should contain information concerning his role in the scene processing as well as his opinion concerning his observations thereof. The criminal investigators, analytical chemists, and prosecuting and defense attorneys use this information to guide their investigations or to prepare their case for trial. The report should state more than a final conclusion of the expert opinion. There should be some narrative explanation of how the expert reached the conclusion.

In some instances, the expert's report will be the only expert evidence presented. The court should be given some explanation of how and why the expert came to his conclusion. A scene report containing a simple summary statement such as: "The items found at 123 Oak Street were consistent with those found at a clandestine lab that manufactures a controlled substance" does not provide other parties involved in the investigation or prosecution sufficient information with which to use to continue with their portion of the investigation.

The previous statement may be a valid conclusion. However, it is too generic and does not provide the reader a sense of what was being manufactured or information to support his statement. A better statement that summarizes the observations made by the scene expert would be: "The items found at 123 Oak Street were consistent with those found at a clandestine

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Table 6.1 Information and Opinion Relationship

Information	Conclusions
Chemical inventory	Used to establish the manufacturing method and the overall production capability of the operation
Equipment inventory	Used to establish the manufacturing method and the per batch capability
Location of the items at the scene	Used to establish the location of each portion of the process at the scene
Original volumes or weights	Used to establish the actual amount of product seized
Seized paperwork	Used to provide a historical perspective of the operation; some operators are detail oriented enough to keep records concerning the percentage yield of each batch
Chemical receipts	Used to provide insight into the amount of chemicals purchased over a period of time

lab that was manufacturing tetra-ethyl-death using the shake and bake method." This wording allows the scene expert to say that in his experience, the items that were observed at the scene were the same as those found in operations that produced a certain controlled substance using a particular manufacturing method. It also provides for the option for alternative manufacturing theories.

A large quantity of information can be derived from the scene. This information can be used to render limited or generic opinions concerning the operation and its capabilities. It will also be evaluated at a later time to establish the particulars of the operation under investigation. Presented in Table 6.1 is a relationship between information that can be obtained at the scene and opinions that can thus be generated.

6.2.2 Laboratory Analysis Information

The information from the scene provides the pieces of the clandestine lab puzzle and a generic outline of how they fit together. Laboratory analysis provides detail for each piece. It fills in the holes and provides answers to any questions generated during the scene investigation. Laboratory analysis supports or refutes the opinions generated at the scene. In some instances, laboratory analysis generates additional questions, requiring supplemental information from investigative sources before a complete opinion can be rendered.

With laboratory analysis, information concerning the identification of the controlled substances being produced as well as the precursor and reagent chemicals located at the lab site is provided. It aids in identifying by-products in the reaction mixtures and the waste products that may be used to establish a particular manufacturing method. Laboratory analysis produces the quantitation data that can be used to calculate the amount of controlled substance present in reaction mixtures and to estimate its production values.

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6.3 Experience and Training

All clandestine lab chemists are forensic chemists. However, not all forensic chemists are clandestine lab chemists. A clandestine lab chemist has the training and experience to render an opinion concerning the existence of a clandestine lab given a certain set of facts. Volumes of information can be generated from a clandestine lab case. The expert trained in clandestine lab matters is able to wade through this to determine which pieces of the puzzle are relevant and which pieces are superfluous.

To be an effective clandestine lab expert, a chemist must have a solid background in basic organic, inorganic, and analytical chemistry. He also should be well schooled in the techniques underground chemists utilize to manufacture a wide range of controlled substances. Finally, he should have access to a variety of analytical databases so that he can cross-reference his analytical information to reduce it to its most logical scenario given all of the known facts. This last is a skill that is only learned over time. In some instances, it is an art based on science and experience.

Presently, there is no formally developed complete course of instruction to train a clandestine lab chemist his trade. Clandestine lab chemists are like the forensic drug chemists of the past. Under ideal circumstances, they serve an apprenticeship under an experienced forensic drug chemist who mentors them in the applications of the analytical techniques of chemistry and forensic science for the examination of clandestine lab evidence. Under less than ideal circumstances, the drug chemist is given a clandestine lab case and is expected to do the analysis and provide the opinions because he is the most qualified (or in some cases the only) chemist available, and by virtue of his education, he is the resident expert.

There are instructional programs that address segments of the forensic clandestine lab investigative process. The Drug Enforcement Administration and the California Criminalistics Institute have programs designed specifically for drug analysis or generic clandestine lab response. These programs only provide the tools that loosely address the issue of the analysis and interpretation of evidence from clandestine labs. Even with this training, the chemist must rely on real-world experience to gain the knowledge necessary to make the tools gained in his basic training effective in his work.

6.3.1 What? How? How Much?

All of the *Who*? *What*? *When*? *Where*? *Why*? and *How*? questions an expert needs to address can be boiled down into three basic questions. The answers to all of the other questions will fall into place if the expert at the scene investigation or the analytical chemist performing laboratory analysis can

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focus on addressing these questions. These three main questions are: *What is he making? How is he making it? How much can he make?*

Answering these questions is not the "be all and end all" of the forensic investigation. The answers generally lead to additional questions that need to be addressed. However, they point the forensic investigator in the direction the investigation should go.

6.3.2 What Is He Making?

On the surface, the answer to this question is relatively easy to ascertain. The analysis of the final product quickly and definitively establishes what the operator was making. Laboratory analyses confirming the presence of a drug or an explosive make a strong argument in favor of one type of manufacturing operation or another.

Establishing the identity of the final product in operational labs that do not contain an isolated finished product is just as simple. The presence of reaction mixtures or waste materials usually will not deter the confirmation of a final product of the operation. Reaction mixtures and waste materials can contain detectable levels of the finished product. The challenge for the chemist is to detect, isolate, and identify the final product in a sample.

At the stage of analyzing waste materials is where the experience and training of the chemist begin to show. There may be only trace levels of a controlled substance. The analytical chemist should be able to recognize the potential final product from the reaction by-products within the mixture. If the proper compound or combination of compounds is present in a mixture, he should devise an analytical scheme that can isolate and confirm the presence of the controlled substance with which they are associated.

Nonoperational labs present a challenge. There are no reaction mixtures or waste products for the analytical chemist to examine. There are no instrumental data on which to hang his opinion and definitively declare the final product of the operation. These situations become mental exercise for the expert. He uses the chemical and equipment information from the scene to identify the most probable product and method used. He evaluates the lists of chemicals that were found at the scene and categorizes them as precursors, reagents, or solvents. He identifies the final products associated with each precursor and the synthesis routes used with each. He then does the same with each reagent chemical. The precursor possibilities are combined with the reagent possibilities, and a hypothesis of the type of operation is derived. To support the hypothesis, the type of equipment is factored into the equation along with any notes, receipts, or additional paperwork with which to complete the puzzle. In Chapter 9, practical applications demonstrate how the chemical inventory can be used to piece together a manufacturing method to answer the question *What is he making?*

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Many chemicals can be used to produce more than one controlled substance. Some can be used for more than one synthesis route for the same controlled substance. With hundreds of different synthesis routes for the various controlled substances encountered in a clandestine lab, it is unlikely that the scene or analytical chemist will immediately recognize unfamiliar chemical combinations associated with obscure chemical reactions. He is less likely to put the two together in the chaos of a clandestine lab scene.

Databases containing the chemicals commonly encountered in clandestine labs can statistically narrow the possibilities. If the chemist has access to this type of database, he can cross-reference the chemicals seized with the controlled substance they are associated with as well as the synthesis route. This information will assist him in objectively looking for a pattern that will indicate what controlled substance the operator was trying to produce.

What else is he making? is a corollary question that should be addressed. As a result of this mental exercise, the chemist should be able to determine what other controlled substances could be made with the combination of chemicals seized from the scene. It is not uncommon for the operator to be experimenting with other manufacturing methods. The expert should not lock onto the most obvious final product. He should expand his evaluation to include or exclude all of the possibilities and any *What if?* questions that could be brought up during the peer review process. Many hypotheses fall by the wayside before a theory can emerge.

The expert must remember his limitations. He is an expert in the clandestine manufacture of controlled substances. Unless he has a Ph.D. in chemistry with an emphasis in organic synthesis, he would be well advised not to speculate about any of the compounds that can potentially be produced using a given list of chemicals. The *What if?* questions in this situation can be endless and beyond the expertise of the chemist, as well as the jurors. Just as the forensic drug chemist is an expert in the identification of cannabis but not in plant identification, the clandestine lab chemist is an expert in the clandestine manufacture of controlled substances, not in organic synthesis. Keep it simple when possible.

6.3.3 How Is He Making It?

The simplest method of answering this *How*? question is to ask the operator or look at his notes. Some operators can be talkative. Others are quiet. In all cases, the operator's statements should be put into perspective, because their culture is one of deceit. However, their statements can be used as a guide for the scene investigation and to corroborate opinions generated from the physical evidence.

Clandestine lab operators do not generally have the education or training to cook without a recipe. The paperwork detailing the manufacturing method

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being utilized is often located somewhere at the scene. This paperwork, combined with the seized chemicals and equipment, demonstrate how the operator was manufacturing his product. However, some operators have committed the process they use to memory, because it is simple, and they have been using it so long. Unfortunately, in these instances, there will be no recipe defining the manufacturing method. Because of this, other forms of corroboration become more important.

The list of chemicals seized from the scene can give insight as to the most likely manufacturing method used. Once the field of possible final products has been narrowed to the most likely candidates, the chemist can compare the list of chemicals required for individual synthesis routes to the chemicals seized from the scene. The synthesis route with the most complete list is the most likely manufacturing method being used. The operator may not have all of the components for the suspected route. However, lack of a complete list of chemicals does not eliminate a synthesis route from consideration. Provided in Chapter 9 are Practical Applications 15 and 16.

As with any opinion, answers to the *How is he making it?* question should only be given after objective review of the physical evidence. However, there are manufacturing methods that are so commonly used and chemical combinations that are encountered so frequently that the on-scene chemist can usually provide a qualified opinion concerning the type of lab and the probable synthesis route. Much beyond that, he is probably treading in water he should not be in, without time to reflect on the totality of the physical evidence. These qualified opinions are necessary to guide the balance of the on-scene investigation. They are used to direct the search for items of physical evidence that will corroborate or supplement the evidence that has been located to that point of time. But, after that, qualified opinions should never be used as defining statements in criminal proceedings. The expert would simply be opening his testimony to cross-examination, if he does not rely on the scientific method to back himself up at all times.

Reaction mixtures and waste materials can provide a wealth of information. Many of these liquids contain all of the information concerning the method used to produce the final product. The precursor and reagent chemical components of a reaction mixture, the reaction by-products in the final product, or waste materials can give information as to the method the operator was using for manufacturing.

The MS provides the clandestine lab chemist a tool with which to identify all of the components within a reaction mixture. Compiled in Appendix K are mass spectral data of reaction by-products that are potentially found in reaction mixtures found in clandestine drug labs. These values are taken from the scientific literature and include the synthesis route associated with each compound. Shown in the table are the compound's five major ions and the

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synthesis routes with which each compound is associated. The chemist must remember that the ion sequence may differ depending on the instrument he is using. If possible, he should run the actual compound to obtain the actual mass spectrum for identification purposes.

The lack of primary standards for the reaction by-products complicates the identification process. The analytic chemist must rely on the analysis of reaction mixtures he synthesized to obtain the mass spectral data of reaction by-products in various manufacturing methods. He should compare these spectra to the spectra in the literature to confirm the identity of the by-products encountered. Instrumental data from nonprimary standards can be used for these identifications, because the identities of these components do not have to be established beyond a reasonable doubt.

The analytic chemist should attempt to reproduce the manufacturing process used by the operator to demonstrate that it actually works. The recipe being used may or may not produce the intended product. Sometimes the reagents called for will not produce the desired effect. Other times, the operator does not have access to the chemicals listed in the recipe, and his lack of knowledge does not allow him to use the proper substitute. The analytic chemist should go through the steps outlined in the operator's recipe to determine whether or not it would function as designed. Understanding the theory of the reaction is one thing. Having direct knowledge as to whether or not the reaction will produce a controlled substance has greater impact in an opinion. The best way to respond to the question *How do you know the operator's reaction will not produce flubber?* is to respond, "I followed the directions found at the location, using samples of chemicals seized from the location, and the result was flubber."

There will be times when the chemical inventory and the laboratory analysis do not provide sufficient information to determine the synthesis route. These instances require the follow-up question: *Why is he using this method?* This question may be a mental exercise that does not have an answer. When all else fails, ask the operator or look at his notes. His level of cooperation may provide the expert the insight he requires. There will be many instances when the expert has to accept the answer: "I do not know."

6.3.4 How Much ... ?

Depending on the size of the operation, the *How is he making it?* question can take a back seat to the *How much is he making*? question. The fact that the operator was making tetra-ethyl-death by the ABC method, at times, seems secondary to the quantity the operation could produce. The amount of finished product seized or that could be produced may or may not affect the type of charge or the sentence that is handed down if a conviction is obtained. The operation's actual or projected production may have nothing

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to do with the manufacturing charges. The accused is or is not manufacturing. The fact or opinion that the operation could potentially produce $10,000,000 worth of drugs or enough explosives to blow up the local police station is probably not an element of the crime. However, it may be used as demonstrative evidence to impress upon a jury the size and scope of the operation.

The three basic variations of the *How much*? questions are as follows:

How much product is there?
How much product could the operation produce per batch?
How much product could the operation produce with the existing chemicals?

These questions may or may not come up at trial. However, some variation of each one will be asked of the expert at some point during the investigation or prosecution of the operator. Therefore, the expert should have the answers to each question.

6.3.5 How Much Product?

Of the three basic "how much" questions, *How much product?* is the most relevant. Many controlled substance statutes use a weight value to establish the severity of the offense. The wording of the statute will provide the analytical chemist guidance in developing an analytical scheme with which to address the legal question of *How much is there?* The wording "…grams of substance…" may require a different analytical approach from the wording "…grams of substance containing…" In either case, the analytical chemist should be able to tell how much of the controlled substance in question was in each sample analyzed.

There are two basic methods of determining amount of substance. The direct method is applicable for situations in which the statutes use wording similar to "… substance containing …." The indirect method is applicable to scenarios in which an accurate accounting of the amount of controlled substance is needed.

The direct method is straightforward. The analytical chemist measures the weight or volume of the substance prior to doing any analytical work. This establishes the weight at the time of seizure. For exhibits in which only a sample was received, the accurate documentation of the original weights or volumes is critical. Without documented weights or volumes, the Court will rely on the only documented value available to them, i.e., the weight or volume obtained by the analytical chemist during his analysis.

With the indirect method, a ratio of the calculated concentration of a sample is used to determine the amount of controller substance in the original item. The amount of substance at the time of seizure can be obtained using

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this calculation. Its use is appropriate when an accurate accounting of the amount of controlled substance is required. The concentration of a sample of the exhibit is determined, and that a ratio of that value is figured into the weight or volume of the original substance. Found in Chapter 9 are examples of how these calculations can be applied.

The resulting value may be subject to interpretation. Issues concerning representative sampling done at the scene, the accuracy and precision of the test methods used to establish the concentration, and the original weight and volume information obtained from the scene will affect the final value.

Proper documentation during every phase of the process is essential. Without supporting documentation, the analysis and the resulting calculations may end up being considered nothing more than speculation and hearsay. If it is not available for peer review, it may not be admissible at trial. However, it may still be useful as an investigative tool.

6.3.6 How Much per Batch?

Determining how much per batch is not a straightforward calculation. There are numerous variables that affect each batch's production. Equipment size, reaction type, recipe, actual versus theoretical yield, and the cook's experience play roles in the operation's per batch production. The expert's training and experience are critical for interpreting the information and factoring in the variables to establish a realistic estimation of the operation's production capabilities.

6.3.6.1 Equipment Limitations

The size of the equipment used in the operation is the major factor that establishes its per batch capability. The operation could have a limitless supply of chemicals, be operated by a Ph.D.-level chemist, and yet still be limited by the size of the reaction flask. A 500 ml reaction flask will only produce a certain quantity of controlled substance during a given reaction cycle.

A simple calculation is used to determine the "per batch" capacity of an operation that utilizes legitimate scientific equipment. As a rule of thumb, the volume of the reaction mixture in a traditional round-bottom reaction flask is two-thirds of its capacity (e.g., a 3000 ml reaction flask has approximately 2000 ml of usable volume). This allows for uniform heat distribution and safe and efficient reflux or distillation. However, because of the operator's lack of technical expertise, the flask may be filled to the top or only 25% of the flask's capacity may be used.

The operator's use of alternative equipment also negates any assumption of proper proportions. There is no rhyme or reason as to why or how full the makeshift reaction vessel is filled. In these situations, as well as the situation in which legitimate scientific equipment is used, the expert should

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rely on the operator's notes to provide guidance as to the per batch production, because operators do not usually deviate from their recipes.

Once the volume of the equipment has been determined, the ratio of chemicals used in the method is factored into the equation. Using the two-thirds capacity guideline, the reaction mixture maximum volume is established. The analytic chemist calculates the amount of precursor and reagent chemicals required to establish that volume. The calculated precursor amount is then used to calculate the amount of product that will be produced with this amount of precursor chemical. Presented in Chapter 9 is an example of how this calculation can be applied.

6.3.6.2 Chemical Limitations

The amount of precursor and reagent chemicals available can limit the per batch amounts. The operator cannot produce more product than the precursor chemicals he starts with allow, no matter what the reaction vessel size. By the same token, the amount of reagent chemicals present will limit the amount of precursor chemical that can be converted into the final product. These values have more relevance in the estimates of the operation's total production capability.

6.3.6.3 Reaction Limitations

In calculating product yields, the expert must decide what value he wants to demonstrate — the actual or the theoretical. The maximum yield of a chemical reaction is theoretically 100% conversion of precursor to product, i.e., 1 mole of precursor chemical will produce 1 mole of product. The actual yield will always be less than the theoretical yield. This number will vary with the reaction, the recipe, and the experience of the operator.

The expert must take into account the difference in molecular weight between the precursor chemical and the final product. The molecular weight of a substance is simply the weight of a single molecule of the substance. The ratio of the molecular weight of the final product and the precursor chemical involved provides a conversion factor that can be used to calculate the amount. In Appendix N, the conversion factors for commonly encountered controlled substances and their associated precursor chemicals are listed. The conversion factor can be used to quickly calculate the weight of a final product from a known weight of precursor chemical, assuming 100% conversion. Practical examples of how to apply these principles are presented in Chapter 9.

The manufacturing methods used in clandestine labs are based on reactions that have been published in the scientific literature. These publications generally report the theoretical and actual yields for the reactions on which they are reporting. Some reactions are efficient and will produce actual yields that approach 90%. The yields of other reactions are substantially less. The

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expert must remember that the published yields may not correspond to those of clandestine operations. The published yields are obtained under ideal conditions, and the conditions of the operation under investigation are usually less than ideal.

The expert should rely upon the percentage yield of the reaction when estimating the amount of final product a given amount of precursor chemical would produce. These opinions should address three situations. First, the perfect situation in which 100% of the precursor is converted into the final product should be addressed. Second, what the literature states the expected yield should be if the reaction were done under controlled laboratory conditions should be considered. Finally, what the yield in a clandestine lab situation would be should be addressed. The analytical chemist who performs the reaction, mimicking the operating conditions of the lab operation under investigation, can obtain the actual yield value. The analytical chemist may compare his values to those of the operator who may have calculated production yields.

The only value the expert can produce with any degree of certainty is the 100% conversion value. This hard value is based upon the molar conversion of a specific amount of precursor to a specific amount of final product, taking into account the differences in molecular weights. The published yield values were obtained under controlled conditions that, as a rule, will not be experienced in a clandestine operation. Thus, those values can only be used as a guide to estimate the production of a given amount of precursor chemical. The yield obtained by the analytical chemist when validating the method under investigation can be used to approximate the operation's yield. However, he should factor in his laboratory technique, the elimination of variables introduced into the operation because of the operator's experience and training, and the "lab" conditions of the operations.

When discussing the yield of an operation, the expert's opinion should simply state that a given amount of precursor chemical would theoretically produce a given amount of final product. He should be willing to acknowledge that the actual value will be lower because of the variables involved in the production of the product. He should also be prepared to describe how he arrived at the lower figures, by the use of published data, of his own analytical experiments, through the use of notes from the operator, or by a combination thereof. Being able to defend his opinion in a calm and organized fashion is crucial to his perceived reliability.

Multistep reactions place additional variables into the equation that should be accounted for. Each step of the manufacturing sequence has a characteristic yield that may or may not be the same as the previous step. In calculating the total production from a given operation, the expert needs to account for the yield estimates for each individual step in the sequence and be able to describe the differences and why they exist, if necessary.

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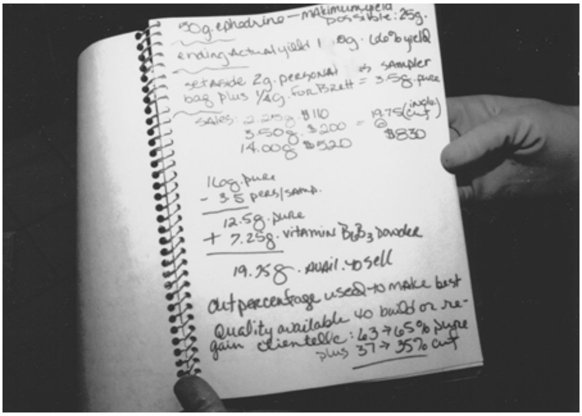

Figure 6.3 Handwritten notes.

6.3.7 How Much per Week?

There are a number of variables that affect an operation's production over a period of time. The synthesis route, the number of batches in the time frame, the cook's experience, and the availability of chemicals will affect this value. Even with these unknowns, there is information available that can be used to produce a historical perspective of the operation's production.

The seized paperwork is the best source of information concerning overall production. The relevant paperwork that can be used to establish production amounts includes sales ledgers, production logs, chemical receipts, and recipes. Such information is generally available at the scene, again because of the typical operator's pack rat nature. Receipts provide a purchasing pattern of the required precursor chemicals. Per batch and per week estimates can be extrapolated from these receipts and other information from the scene to obtain a historical production pattern or to project one into the future.

Some operators do the work for the expert. They have been known to document the per batch production, some to the extent of even calculating yield percentages (Figure 6.3). Other paperwork found at the scene may document sales or distribution information. If the documents contain dates associated with amounts, a historical portrait of the operation may be obtained. The key to using this type of information is the ability to decipher the operator's shorthand or codes. This situation is an example of where the expert's knowledge of clandestine operations is essential. His ability to translate cryptic notes into understandable language assists investigators and attorneys involved in the case by providing a better understanding of the various portions of the operation to which the notes refer.

The operator's recipes can also be extrapolated to provide a historical representation of the operation's production. However, in doing so, a number

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of assumptions must be made. Operators do not tend to deviate from the recipes they use. Therefore, per batch estimates can be calculated; the amount of precursor chemical denoted can be used to estimate a time frame for their consumption. Any estimates beyond that will be considered speculation on the expert's part without additional information.

Assumptions come into play in this situation, and the numerous *What if?* questions can be asked. The expert can calculate the single-batch production using the techniques listed above. The number of batches per day, per week, per …, is subject to conjecture without additional information. These estimates can be made, but the variables used to provide the opinion should be made up front. For example, at 100% conversion, 1000 g of ephedrine hydrochloride will produce 920 g of methamphetamine hydrochloride. It is misleading for the expert to claim that the whole 920 g could be produced at once if the operator's recipe called for 10 g of ephedrine. All of the variables must be reasonably addressed. With the per batch information factored into the opinion, it would take 100 batches to convert all 1000 g of ephedrine into methamphetamine. More information is needed to determine how long that would take, or another assumption would have to be made and presented to qualify the opinion.

It is in the best interest of the expert to be candid about the information used to produce his opinion. Playing word or number games in trial or deposition can compromise the expert's credibility or diminish his objectivity in the eyes of the jury. He must remember that his role is to provide the information needed for jury members to make informed decisions. Providing information concerning the assumptions used to make the opinion will reduce the *What if?* questions that can be posed by either counsel. Using the previous example, the expert would be wise to say that, assuming 100% conversion, the operation could theoretically produce 920 g of methamphetamine in 100, 10-g batches.

If more detail is requested, more information is required, or more assumptions must be made. *How long does a single batch take?* and *How long between batches?* are not unreasonable questions. Their answers will affect the span of time necessary to convert the entire amount of known precursor into the finished product. The expert should not render an opinion concerning production time frames without qualifying his response by establishing the parameters that frame it.

6.3.7.1 Production with Available Chemicals

How much controlled substance could the operation produce at the time it was seized? This question can be answered by using the chemicals that the suspect had on hand to estimate the amount of product that could be produced. To provide a total picture of the operation's production potential, the expert will

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need to calculate the amounts using the most-abundant and least-abundant chemicals. The most-abundant chemical calculation will provide information concerning the operation's potential, if the balance of the necessary chemicals is obtained. The least-abundant chemical provides information concerning the limitations on the amount of product the operation could produce at the time the operation was seized. The focus should still be the operation's potential product. The time requirement does not enter into these calculations. Obviously, depending on whether the expert is testifying for the prosecution or for the defense, there will be differing emphasis placed on cross-examination. It is always better to have all of the information, either way.

These calculations differ slightly from the "per week" estimates in that the expert takes into account the amount of reagent chemicals required. If the operator does not have the necessary reagent chemicals, the reaction cannot take place. For example, the amount of methamphetamine that can be produced from the 1000 g of ephedrine from the previous section is zero if there are no reagent chemicals to facilitate the conversion. That is not to say that a clandestine lab does not exist. It only means that at the time of the seizure, the operation could not produce methamphetamine.

In establishing the chemical ratios required for these calculations, the expert should rely on the operator's notes or recipes. These will provide the most accurate information concerning the operation's production methods, which is used to estimate the operation's production potentials. Not having access to this information, the expert should fall back on ratios from clandestine operations using similar manufacturing techniques. If these sources are unavailable to the expert, the scientific literature should be consulted.

The expert should provide the 100% conversion value as well as an adjusted yield value using available percentage yield values. If these are unavailable, he should stick to the 100% yield value, acknowledging the fact that the actual value will be lower. As with all production estimates, the expert needs to acknowledge that the actual production will be less than the total conversion value.

6.4 Summary

The clandestine lab investigator must answer the *Who? What? When? Where? Why?* and *How?* questions concerning the operation. The forensic clandestine lab investigator will be most concerned with specific questions of *What? Where?* and *How?*

- The following are important *What?* questions:
  - What were they making?

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- What chemicals and equipment were used in the operation?
  - What production methods were used?
- The following are important *Where?* questions:
  - Where was the lab located?
  - Where were specific parts of the lab located?
  - Where was the finished product or waste material located?
- The following are important *How?* questions:
  - How was the operator making the controlled substance?
  - How much finished controlled substance was there?
  - How much finished controlled substance could the operator make?

To answer these questions, the expert requires information from a variety of sources in order to form a strong objective opinion. The information can come from the scene of the operation, from the laboratory analysis of samples taken from the scene, or from the expert's specialized training and experience in the area of the clandestine manufacture of controlled substances. The information from three sources is combined to formulate the total picture of the clandestine operation.

Information to answer the *What is he making?* question can be obtained from the seized notes and recipes, the lab operator, the laboratory analysis of samples from the scene, or the chemical inventories. Information to answer the *How is he making it?* question can be obtained from seized notes and recipes, the chemical inventory, the equipment inventory, or the laboratory analysis of samples from the scene. Finally, information to answer the *How much…?* questions can be obtained from the seized notes and recipes, the laboratory analysis of samples from the scene, or the chemical inventories.

The opinions provided concerning the existence of a clandestine lab should be neutral and based upon the known facts. As with all forensic evidence, it should be presented in an objective fashion that allows the judge or jury to make an informed decision based on objective information.

The expert opinions provided in the investigations and prosecutions of clandestine labs are key to determining the direction of the investigation or the subsequent trial. The expert can imply whatever he deems reasonable, i.e., that the operation was the largest ever seized. He may, on the other hand, reduce the significance of the same set of facts to diminish the seizure to an insignificant occurrence.

The forensic expert must remember that his purpose is to evaluate the evidence in an objective manner and provide his opinions in an understandable fashion. He is not in Court to establish guilt or innocence. The purpose of an expert opinion is to assist those charged with establishing guilt or innocence by providing the information they require to make an informed decision.

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7 Testimony

The analysis of evidence submitted to forensic laboratories by police agencies has often been the focus of relevant articles published in the scientific literature. Presentation of these results to a jury is just as important but receives little notice. If the information is not relayed to the trier of the fact (i.e., the judge or the jury) in an understandable fashion, it may be ignored.

The presentation of forensic evidence to a jury involves conveying technical information to a group of people who may have little to no knowledge of the subjects under discussion. The expert's task is to present his information in such a way as to inform, without insulting, the broad range of personalities found in a jury. Educational levels will certainly vary, but more likely is that few will have any knowledge whatsoever about clandestine production of controlled substances. The expert's testimony, therefore, plays a key role in educating a judge or jury about what a clandestine lab is and how the evidence does or does not indicate that one exists in the particular case before them.

In this chapter, the focus will be on the format that courtroom presentation of forensic evidence in a clandestine lab case should take to be most productive. Chapter discussions will be directed toward forensic chemists. However, these principles can be applied by anyone involved in presenting physical evidence.

The expert's testimony is an essential element in the prosecution of a clandestine lab. Investigators gather the pieces of the clandestine lab puzzle, collecting the facts that establish who the participants in the illegal activity are and delineating the items of evidence that were seized. The expert's explanation puts the pieces of the puzzle together. His description of how the chemicals and equipment can be combined to manufacture a controlled substance is critical in establishing that a crime was committed. If the expert does not effectively relay this information to the jury, a conviction may be unnecessarily difficult to obtain.

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There are two situations in which a forensic expert may be called to testify in a clandestine lab case. The first situation is where the expert was an active participant in the lab seizure or performed laboratory analyses on the evidentiary samples. In the other situation, the forensic expert acted as an independent expert who evaluated the information concerning a suspected clandestine lab operation. Although each situation is handled in a similar manner, there are subtle differences.

7.1 Case Preparation

Trial preparation for a clandestine drug lab case begins long before the trial or deposition subpoena is issued. Conversations with the investigator preparing an affidavit for a search warrant are important. Every comment or opinion the expert gives to the investigator can potentially have evidentiary value. His expert opinions are used in the affidavit to justify the search warrant or guide the investigation in one direction or another. If he provides incorrect or faulty information, the search warrant could potentially be invalidated, or the investigation could go into an ineffective or inappropriate direction.

The expert providing opinions to investigators or attorneys concerning the potential existence of a clandestine lab should document his conversations, noting with whom he spoke, any facts that were presented, and the opinion he provided. The technical nature of the information the expert provides to a nontechnical individual has the potential to be inaccurately reflected in documents that are submitted by others to the Court. Therefore, it is wise for the expert to maintain files documenting and, hopefully, clarifying these conversations.

What the scene chemist does and says at the clandestine lab scene should also be documented. As the on-scene technical advisor, his words and actions guide the investigation. Even if he is not the person who actually finds or seizes a particular item of physical evidence, his opinion is the basis for the investigator's actions. Everything the scene chemist does and says at the scene has potential evidentiary value. The chemist determines what samples are taken, what items should be disposed of due to contamination, and what controlled substances were potentially being manufactured.

A good photographic record supplemented with comprehensive notes taken at or shortly after the search will be invaluable come trial time. These records will allow the chemist to remember details of that particular lab operation and the sampling procedures used on which he based his opinion of the existence of a clandestine laboratory operation. Photographs and notes will also help the chemist remember why certain items were sampled and others were not.

Generically, the laboratory analysis and testimony concerning samples from a clandestine lab are no different from the analysis necessary to identify

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a controlled substance. A reaction mixture is, in essence, a liquid in which the chemist is trying to identify any controlled substance; he also can use this to identify precursors, by-products, diluents, reagents, and solvents. Many of the same techniques used to identify the controlled substance are simultaneously used to identify these other components.

The generic chemist's testimony concerning the analytical results is as straightforward as the analysis. The chemist describes how and when he received the sample, his examination procedures, and finally, his results, all of which should be supported by his working notes. A standard set of questions is generally encountered, and a great deal of trial preparation is not required for the experienced chemist.

The clandestine lab chemist takes the analysis of clandestine lab samples beyond the basic identification of a controlled substance. The required opinions necessary to establish the elements of clandestine manufacturing mandate the identification of the other components of the various mixtures found at the scene. The chemist should devise his analytical scheme in anticipation of presenting his results to a jury. Opinions that are generated concerning the operation or a specific exhibit must be substantiated by the analytical chemist's laboratory data to establish the facts beyond a reasonable doubt. If the analytical chemist is going to talk about a specific chemical within a mixture, he should have the analytical data to support its existence. If the analytical chemist is going to talk about the synthesis route used to produce the methamphetamine detected in the reaction mixture he analyzed, he should also have identified the components in the mixture that support his conclusion.

The clandestine lab chemist's trial role significantly deviates from the traditional forensic chemist's role by providing opinions concerning the "how" and "why" of the operation. Traditionally, the forensic drug chemist's testimony does not include significant speculation concerning the condition or potential use of the exhibit he examined. The clandestine lab chemist's testimony can potentially be filled with opinions concerning the various aspects of clandestine lab chemistry. With the information from the laboratory analysis and the scene information, the chemist should be able to form opinions concerning the following: the synthesis route being used, the exact step in the synthesis at the time of seizure, other synthesis routes that may have been used, an estimate of the production of each synthesis route using the chemicals and equipment on hand, and finally, determination of the total amount of finished product. Each of these opinions needs to be supported by some type of objective physical evidence.

The chemist's courtroom presentation of a clandestine lab case is the other half of his job, and in some instances, that may be the most important. His testimony ties all the pieces of information together and must be done in an understandable fashion. His presentation of the technical information

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is a key factor in establishing the cause and effect relationships between what appear to be ordinary items and the manufacturing of controlled substances. A conviction may be difficult to obtain if the chemist cannot demonstrate how it all fits together, no matter how much evidence is presented.

The chemist's courtroom presentation can be broken into two distinct phases: the pretrial conference and the actual testimony. This discussion will be from the perspective of a generic expert witness. Although the prosecution presents most of the expert testimony in clandestine lab cases, the defense has the opportunity to present its own expert's opinion of the significance of the physical evidence. Whether the chemist is testifying for the prosecution or the defense should be a moot point, because in either case, the expert should be objectively evaluating the evidence and, only after careful consideration, relaying his opinion. Although the steps in the process for either side are essentially the same, it is obviously in the interpretation of the evidence and formation of an opinion that differences may arise.

7.2 Pretrial Conference

A pretrial conference with the attorney should be scheduled as soon as the chemist knows he will be testifying in a clandestine lab case. Ideally, the attorney handling the case will be knowledgeable in clandestine lab prosecutions. In the real world, prosecutors handling these cases are often inexperienced and not knowledgeable of their intricacies. Because of the special nature of clandestine lab cases, defense attorneys may not be any better prepared. Therefore, the chemist's first job is that of a teacher.

During this pretrial conference, and all subsequent meetings, the chemist should educate the attorney about clandestine drug labs in general; educate the attorney about the specifics of this clandestine drug lab; explain what indicates that a clandestine drug lab exists in this instance; determine what items are missing; determine the sampling procedures that were used; and finally, explain the chemical disposal process (if used).

7.2.1 Educate the Attorney about Clandestine Drug Labs in General

The chemist should explain to the attorney exactly what a clandestine lab is and delineate the many forms it may take. The attorney must truly understand that clandestine labs come in many shapes and sizes. The chemist must explain what they are and, just as importantly, what they are not. The attorney must understand that clandestine drug labs can range from a simple "crack" conversion operation performed in a kitchen to an elaborate chemical synthesis using exotic chemicals and expensive equipment. He must be informed

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that the common household equipment and the proper combination of chemicals that can be found in "his" own house can be turned into drugs or explosives. However, the totality of circumstances surrounding the case will determine whether a clandestine lab exists or not.

Auxiliary issues concerning clandestine labs should also be addressed in the initial interviews. Information concerning the direct and indirect hazards related to the operation should be relayed. The prosecutor must realize that the significance of the case goes beyond manufacturing the operation's final product. He must understand the jeopardy to the health and welfare of everyone who had contact with the operation. This may not be a significant point in proving the facts of the case. However, it may place a different spin on the overall significance and priority of this type of case.

7.2.2 Educate the Attorney about the Generalities of this Clandestine Drug Lab

Once the attorney knows and understands what a clandestine lab is, the chemist can explain what type of operation is being dealt with in this particular instance. The chemist should begin by generally explaining the synthesis route he suspects was used in the operation and what chemicals and equipment were needed. He should walk the attorney through the process, providing a step-by-step explanation of how all the items fit together. This big-picture overview should be done in a generic way, in order to paint a picture of how the operation would be conducted in a perfect world.

7.2.3 Tell the Attorney what Indicates that a Clandestine Drug Lab Exists in this Instance

The chemist can explain the specifics concerning which items of evidence support the opinion about this operation, once the attorney understands the process being used. At this point, the chemist shifts his focus from the ideal situation to the case at hand. He may explain how the reaction flask and heating mantle in an ideal scenario are an ordinary Mason jar and pressure cooker, respectively, in the present case. During this portion of the interview, he explains how each piece of physical evidence fits together to complete the manufacturing operation puzzle.

7.2.4 Tell the Attorney what Items Are Missing

Crime scene puzzles are always missing pieces. Clandestine lab scenes are no different. The chemist must inform the attorney what items were not found at the scene, what their significance to the process was, and if their absence affects the opinion about this operation. If there is a problem justifying the existence of a clandestine lab through the physical evidence (or lack thereof), the attorney should be made aware of it.

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The chemist is not there to prove the attorney's case by manipulating insufficient physical evidence to give the illusion that something exists. He is there as an objective seeker of scientific truth. If the case is legitimate, the physical evidence is there. The chemist identifies what pieces of the puzzle are missing and what could possibly have been substituted. Again, if the evidence is not there, the chemist should make the attorney aware of what is missing and what is needed to fill the hole.

7.2.5 Explain the Sampling Procedures that Were Used

The attorney needs to understand the sampling scheme used at the scene and the thought process behind it. He must understand that it is unrealistic to sample every container at the scene. Therefore, the chemist must explain the purpose behind the sampling process and how this scientific method has brought calm to chaos through the condensing and consolidating of the mass of physical evidence into manageable packages. With this process, everything possible is done to preserve the integrity of the scene and to document all of the physical evidence that was located. The attorney must understand the thought process behind the determination of which items were sampled and how each sample was selected to serve as a piece with which to complete the puzzle. The attorney must be able to trust and rely upon the training and experience of the scene chemist.

7.2.6 Explain Chemical Disposal (if Used)

The toxic and hazardous nature of many of the chemicals and equipment seized from a clandestine lab requires disposal according to protocol by following guidelines mandated by an established set of local, state, or federal regulations. The lack of proper storage facilities has led many jurisdictions to opt for more immediate disposal after proper sampling and photography have been completed. The attorney needs to clearly understand that such protocol exists for the safety of the personnel at the scene, for lack of proper storage facilities of the seized chemicals, and for avoidance of contamination of the courtroom by bringing hazardous material in during trial. It must also be stressed that no evidence was destroyed. It is, therefore, critical that all evidence be documented and photographed and only then sampled. Original volume must also be noted and photographed before material is disposed of according to the appropriate regulations.

7.2.7 Outline Testimony

Trial attorneys follow a basic rule concerning questioning: do not ask a question unless you know the answer. There is no reason the chemist should be surprised by a question from the attorney whose client he is representing.

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The chemist should work with the attorney to create a known line of questioning for the direct examination. It should flow smoothly with no surprises, with all relevant questions being addressed. Presented in a Practical Example in Chapter 9 is a scenario of shaky testimony resulting from incomplete testimony preparation.

During this part of the pretrial process, the chemist and the attorney should develop an understanding concerning their respective limitations in each other's fields of expertise. Each should have enough knowledge of the other's job to be helpful, yet understand and accept that such knowledge can be dangerous in less-experienced hands. As the number of cases each attorney and chemist present at trial increases, their knowledge and comfort levels will increase. However, each should know and understand separately or together his limitations and particular role in the process. The chemist should present his knowledge of the relevant statutes concerning the manufacture of controlled substances, simultaneously not attempting to make legal assumptions without consulting the attorney. The attorney should never make assumptions concerning certain pieces of physical evidence as scientific fact without consulting the chemist.

For example, while attorneys are masters of the English language as it relates to the law, their expertise dwindles to the apprentice level when English is applied to the technical arena of forensic science. In this instance, the chemist's knowledge of the proper terms should be utilized to create a script of questions that will present the evidence in a logical sequence that uses the appropriate terminology. This provides the chemist with knowledge of what the question is, and the attorney with the knowledge of what the response will be, simple yet essential.

A word of caution is necessary for trial. It is important for both sides to stick to the script. Slightly changing the way a question is phrased may elicit a response that is not expected. At the same time, an unexpected response may result in additional questions to explain what should have been apparent if the original answer was presented as planned. Again, it is always dangerous to play with the unknown, and this can give opposing counsel grounds to challenge the testimony that really counts. The strength of expert testimony may lie simply in how well conversant in his subject the judge is or how the jury perceives the expert.

How the exhibits will be presented should be discussed. It is not wise for either party to see the exhibits for the first time in the courtroom. The chemist and the attorney should discuss what exhibits demonstrate the points they want to make. At the same time, they should develop a logical presentation sequence that will relate each item's use in the operation to the jury. This type of preparation provides a flowing and understandable format for the testimony that does not surprise either party.

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7.2.8 Discuss Visual Aids

The use of simple, concise visual aids should be discussed with the attorney. Used properly, visual aids can take a complicated process and reduce it to simpler terms. The combination of oral explanations with visual reinforcement provides a more interesting testimony format and enhances retention of the information presented. For example, visual aids can be used to demonstrate what items are necessary to manufacture a controlled substance and to compare this information to the actual evidence exhibits. They can also be used to demonstrate how the equipment exhibits fit together to make the reaction apparatus.

7.3 Testimony

The testimony of the chemist or forensic expert can make or break a case that is presented to a jury. The expert's appearance, demeanor, and presentation affect how jury members interpret the information they receive. Lack of testimonial skills or an improper attitude or presentation can substantially reduce the credibility of even the most qualified expert. During his presentation, the chemist should present a professional appearance, tell the truth, address answers directly to the jury, and answer only the questions presented.

7.3.1 Direct Testimony

Clandestine lab forensic testimony is divided into three basic phases. The level of expertise and the number of people involved in the forensic portion of the investigation will dictate the number of people required to testify. The three phases of testimony include scene processing, analytical examination, and expert opinions.

In many situations, the scene chemist acts only as a technical advisor during the seizure of a clandestine lab. He is not responsible for the seizure of the physical evidence and may not be called to testify. However, he provides the expert opinions and the technical knowledge used to guide the investigator's decision to seize or not seize a given item. As such, the scene chemist should be able to articulate the thought process he used during the processing of the scene, if he is called to testify. He should also be able to articulate his role in the chain of custody of the items and samples seized into evidence.

The way testimony is given by an analytical chemist in a clandestine lab case will be similar to the way testimony is given about any controlled substance. The voir dire process, the testimony establishing the chain-of-custody, and the results of the laboratory analysis are essentially the same. The level of a chemist's expertise in the clandestine manufacture of controlled sub-

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stances will determine how far beyond the identification of the sample components his testimony may be allowed to extend. In some instances, he may be able to articulate how the laboratory results can be used to outline or depict the role of the samples in the manufacturing process. In other instances, his lack of knowledge would dictate that someone who specializes in clandestine manufacturing techniques would be more qualified to address those issues.

Expert testimony concerning how seized items could have been used to manufacture a controlled substance should be the dynamic part of the chemist's presentation. This is the pinnacle of the expert testimony and should be presented in such a way as to relate technical information in a nontechnical format that is not condescending. This testimony should fit all of the pieces of the puzzle together. The chemist or forensic expert links the testimony concerning the scene processing to the analytical results, hopefully painting a picture of how the manufacturing process worked. The chemist should interact with the appropriate exhibits and prepared visual aids to demonstrate to the jury how the exhibits relate to each other.

There are a few basic principles that should be followed, as they apply universally to the "success" of expert testimony — the witness' appearance, how he answers questions, how he addresses the jury, and how he interacts with exhibits.

The forensic expert should present a professional appearance. He should dress in business attire that is appropriate for the region of the country and the court in which he is testifying. While a sport coat, tie, and cowboy boots may be acceptable in some state court situations in the western United States, this manner of dress would be distracting in a federal court in Manhattan. He should address the attorneys representing each side with the same respect. He must clearly present himself as being there to establish the scientific facts of the case, not being there as a hired gun for one side or the other.

The chemist should direct his answers to the jury members. His testimony is for their benefit. Answers to questions should be presented in terms that the layperson can understand. Obtuse technical language should be avoided. However, when technical jargon is required, it should be explained in a noncondescending manner.

The chemist must adjust his testimony style to facilitate the job of the court reporter and, hence, the official written record. During his testimony, he must refer to evidence items by their exhibit number. Even though he is holding the exhibit, and the jury can see what is being described, he must always be aware there is a written record being taken. The written transcript must reflect his actions as well as his words. The reader of the transcript would not understand "Insert this here" but would comprehend "Place Exhibit #13 into the outlet on Exhibit #24." Therefore, the expert should

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always describe what he is doing with the evidence items so the written record will accurately reflect his actions.

The attorney's style, the personality of the chemist, and what the Court will allow dictate the format used for testifying about clandestine labs in general and about the specifics of the particular case. Testimony formats range from questions with rambling narrative answers to questions with specific responses. The chemist and attorney should work together to establish a basic approach, so they will both be comfortable with this portion of the testimony. If the chemist is not comfortable giving narrative answers in front of the jury, his testimony may be more effective with a specific-answer format.

In the narrative-answer format, the attorney asks an open-ended question. The chemist then provides a narrative answer covering as much of the topic as appropriate. When necessary, he asks the Court's permission to use the exhibits or prepared visual aids to demonstrate his answer to the jury. This method of testimony can be entertaining as well as informative. Such information may have more jury impact because of the deviation from the typical, dry question and answer format.

In the specific-answer format, specific questions are asked and specific answers are given in order to present information to the jury. This format is drier and can be more time consuming than the narrative-answer approach. The jury may get bored, lose interest in the testimony, and end up not retaining key information concerning the facts of the case. Still, there are situations in which specific questions with a yes or no response should be used to introduce large amounts of boring material. Alternating narrative answers with quick yes and no answers is also a more effective way to keep the jury's attention during lengthy testimony.

There may be occasions where, regardless of how prepared the chemist and attorney are for a narrative-answer format, the opposing counsel may object and the Court may rule that only a specific-answer format can be used. It is, therefore, essential that the attorney be knowledgeable in the particular type of clandestine drug lab being tried so that appropriate questions may be substituted at a moment's notice.

The points the opposing counsel would normally address should be covered during the direct examination. This is done to avoid an appearance of deception. Examples of these points are as follows: "What are the legitimate uses for the chemicals and equipment seized from the scene?" "Why were the chemicals and equipment disposed of?" "What other products could result from the combination of chemicals that were seized?" Addressing issues like these keeps the attorney focused on objectivity and transparency. Addressing questionable issues during direct examination also takes the wind out of the opposing counsel's sails by addressing them in an up-front manner.

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There are situations in which enough evidence was obtained to indicate that a particular synthesis was being used, even though all of the chemicals or equipment necessary for a particular synthesis route were not found at the scene. In these instances, the expert's testimony should address this issue during the direct examination. The opposing counsel will contend that the expert cannot make the determination of the existence of a clandestine drug lab, because all the necessary items were not found at the scene. If the issue is addressed immediately during direct examination, and the expert is confident with his opinion, he is less likely to get caught in the "What if?" game the opposing counsel will try to play in order to establish "reasonable doubt."

7.3.2 Cross-Examination

Cross-examination methods used to question experts vary widely. The options can range from "No questions" to a wide range of hypothetical questions designed to cloud the issues raised during the direct examination. There should be no problem with the cross-examination if the expert is properly prepared for direct examination.

During cross-examination, the expert should keep in mind the following: be sure to understand the question, answer only the question, do not argue with the attorney, know his limits, and be truthful.

Understanding the question and answering only the question that is asked are closely related issues. Attorneys that specialize in litigation are often wordsmiths who ask questions in a particular way for a reason. They craftfully word questions in a specific manner to elicit a specific response. The question may be intentionally vague and unclear so as to obtain a particular response from the witness in an effort to undermine his credibility. The expert should not outfox himself by playing word games with a professional wordsmith. Answering what he thinks the question is or what he thinks the attorney wants to hear will only lead to problems and misunderstandings. If the expert does not understand the question or does not like the way it is worded, he should ask the attorney to repeat or rephrase it. However, if the expert thinks the response to a particular question would be intentionally misleading, he should preface his response with a brief explanation. All of this can be cleared during redirect examination; however, the chemist's credibility may already be compromised and a certain amount of doubt already created.

Do not argue with the attorneys. Opposing counsels have been known to badger and harass expert witnesses. This combative approach to cross-examination is done to shake the witness' confidence or fluster the witness into giving inaccurate answers to his questions. The opposing counsel should be treated in the same courteous manner as the attorney the expert is representing; no appearance of favoritism should be visible on the part of the expert. If the expert loses composure, a certain amount of credibility may be

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lost. A calm, composed response to an offensive line of questioning can give the jury the impression that the expert is being abused by the opposing counsel, potentially giving the testimony more credibility.

The expert should know the limits of his expertise. Even if he possesses knowledge beyond what the common juror would, the expert would be well advised to limit his testimony to the areas in which he is strongest. A degree in chemistry does not make the chemist an expert in all areas of science. If the answer to a question is beyond his expertise, he should simply say so. There is no sin in defining the scope of his knowledge. Stating facts without direct knowledge, embellishing the truth to make a point, or showing off his expertise could all end up causing more harm than good.

Telling the truth to the best of one's knowledge is the best defense to an attack on your credibility. Experts work long and hard to build reputations as experts; one bluff or exaggeration can forever tarnish credibility.

7.3.3 Independent Expert

There are times a qualified chemist or forensic expert will be called to provide testimony concerning a suspected clandestine lab in which he had no direct involvement. A chemist or forensic expert familiar with clandestine labs can give expert testimony concerning his interpretation of the facts of the case. The independent expert takes similar steps in preparing for this type of case as if he had direct involvement in the case.

The pretrial conferences with the attorney cover essentially the same areas as the pretrial conferences held when the expert was an active participant in the lab seizure. His review of the case documentation may bring up additional questions that were not addressed during the original investigation. The independent expert may also ask for additional examinations to be performed or additional items of information to be provided.

The independent expert's testimony is almost solely opinion, giving a little more latitude to what he can testify to. He must stick to the facts of the case, but, as an expert giving an opinion, he is allowed to make some assumptions based on those facts, being careful not to exaggerate the significance of a point. The independent chemist should be conservative in his opinions, keeping in mind that the goal is the truth.

Many of the items seized in clandestine drug labs have legitimate uses, and the independent expert should be willing to admit to such uses. If the facts as presented indicate that the items were being used to manufacture a controlled substance, he should be willing to say so. By the same token, he should be willing to accept legitimate reasons for the combination of chemicals and equipment, if the totality of circumstances dictates them. The independent expert is not a hired gun; he is there to present all of the options to the Court to allow for a fully informed decision to be made.

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7.4 Visual Aids

The use of visual aids is an important component of the trial presentation of a clandestine lab case. The use of visual aids will allow the expert to demonstrate the relationships among items that helped him form the opinion that the items were used to manufacture a controlled substance. Studies show that an audience will retain approximately 55% of what they see as opposed to 10% of what they hear. Thus, the use of appropriate visual aids should enhance a point the expert is trying to make. Such a courtroom change will also break up the dull question and answer format of a trial, leaving a greater impression on the jury.

The expert can use visual aids as memory refreshers when talking about a process or a particular set of exhibits. A well-prepared set of visual aids can be used as an outline for a narrative testimony. The expert can use the key words and items the jury must remember to remind him of what he wants to say.

To be most effective, visual aids should be simple, easy to read, easy to understand, and colorful.

7.4.1 Simple

Visual aids should be simple. They should be prepared using the one-to-one rule, i.e., one central idea per visual aid. If a visual aid is too busy or confusing, the point may be lost in the clutter, thus resulting in the jury ignoring it. Shown in Figure 7.1 is an example of a busy visual aid. It presents a lot of relevant technical information concerning conventional serology testing in one package. All of this information may be informative to the jury. However, what single piece of information does the expert want the jury to retain? Does he want to correlate the number of positive tests between the suspect, victim,

RESULTS OF A BLOOD STAIN EXAMINATION

Sample	ABO	PGM	EsD	GLO	EAP	ADA	AK
Suspect	O	1+1-	1	2	B	1	1
Victim	O	1+1-	1	1	B	1	2-1
Sheet	O	1+1-	1	1	B	1	2-1

Frequency of occurrence (victim) 3:10,000

Frequency of occurrence (suspect) 63:10,000

Figure 7.1 Busy visual aid.

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RESULTS OF A BLOOD STAIN EXAMINATION

Victim Blood = Sheet Stain ≠ Suspect Blood

Frequency of occurrence (Victim/Sheet) 3:10,000

Frequency of occurrence (Suspect) 63:10,000

Figure 7.2 Less busy visual aid.

and evidentiary sheet? Does he want the jury to know the frequency of occurrence each set of tests represents? Does he want the jury to remember that the stain on the evidentiary sheet was the same as the blood of the victim and not the suspect?

In designing the visual aid, the expert should ask himself, "What single piece of information do I want the jury to retain?" From there, the visual aid can be easily designed. Complete sentences should be avoided unless absolutely necessary. Too many unnecessary words create a busy visual aid. Key words, phrases, or ideas that emphasize the expert's opinion and will stick in the jury's mind should be used. Shown in Figure 7.2 is an example of how the information from Figure 7.1 can be simplified to demonstrate the relationship between the victim's blood, the stain on the evidentiary sheet, and the suspect's blood. The visual aid simply states the expert's opinion that the victim's blood and the stain are the same. It supplements that conclusion by adding statistical information concerning the frequency of occurrence that would produce the test results. The information concerning the results of the individual tests used to make the conclusion is unnecessary detail that may detract from the central point being made.

7.4.2 Easy to Read

The expert should put himself in a juror's position and determine whether everything on the visual aid can be seen. Points that cannot be seen clearly will not receive the visual reinforcement desired and will lose their impact. Easy to understand terms and symbols should be used. Again, key words and phrases in large bold print produce a good visual impact.

7.4.3 Easy to Understand

Technical symbols and abbreviations should also be avoided unless they are explained and will result in ease of comprehension. The expert should also

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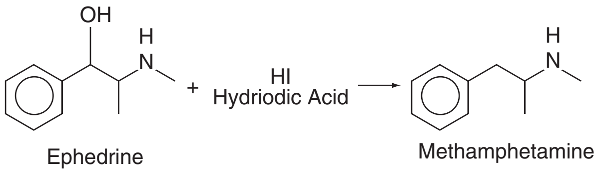

Figure 7.3 Methamphetamine reaction mechanism.

stay away from the use of chemical structures and formulas. Most jurors do not have an extensive science background. However, the expert can use the jury's basic knowledge of chemistry to remember a chemical. An example would be hydriodic acid. For the nonchemist, the word is hard enough to pronounce, much less spell. If the expert explains the use of the abbreviation "HI," he may provide the jury with an easier way to remember the chemical.

Many chemicals have some type of abbreviation or common name to which the jurors can relate. The expert should utilize as much common terminology as possible in his visual aids and his narrative explanations. The same philosophy holds true with diagrams of reaction apparatuses; the diagrams should be as simple and generic as possible, while still getting the point across.

In Figure 7.3, a simplified version of the reaction mechanism for the reduction of ephedrine or pseudoephedrine into methamphetamine utilizing hydriodic acid is presented. It follows the simplicity guide from above in that it shows that a group of chemicals reacts to form a controlled substance. The downside to this example is that the chemical formulas used to make the point may be intimidating to a layperson. Some jurors may subliminally block any information that uses this format, even though it is presented in its simplest form.

In Figure 7.4, a simple list is presented as an alternative to the technical diagram in the previous figure. The list presents all the chemicals required to manufacture methamphetamine with the HI reduction technique, using the chemical's common household name. Presented properly, this list can be used to demonstrate the same concepts relayed in Figure 7.3 in a visual manner that will keep the attention of some of the jurors.

7.4.4 Colorful

Color can be an effective tool, especially if a single visual aid will be used to make a number of different points. While each color represents separate ideas or concepts within a single visual aid, the one idea per one visual aid concept is maintained. An example of this would be to group the list of chemicals in Figure 7.4 by their place in the manufacturing process. The chemicals required for each step of the process could have a distinct color associated with them [i.e., ephedrine, HI, and phosphorus would be in red (Step 1); lye

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# 2	Ephedrine
# 17	HI Phosphorus
# 8	Lye
# 37	Freon
# 22	HCl

Figure 7.4 Simple list.

would be written in blue (Step 2), etc.]. In a different color, the exhibit number could be placed next to the corresponding chemical, indicating which chemicals were present at the lab scene. (Numbers in the left column would be written in black.)

In this situation, the expert could use the visual for more than one purpose. First, it provides a complete list of the operation's chemical requirements. Second, it lays out the sequence in which the chemicals are used. Finally, it directly relates the chemicals required to manufacture the controlled substance in question to the items that were located at the scene. The different colors distinguish the different concepts involved. Therefore, this single visual aid can be used to emphasize three separate but interrelated ideas. These two additional ideas can be presented on the original visual aid, but they are differentiated by different colors.

7.4.5 Types of Visual Aids

The types of visual aids that may be used in court are photographs, slides, flip charts, or evidence exhibits. More often than not, some combination of these is used during the trial.

7.4.5.1 Photographs

Photographs are one of the most commonly used types of visual aids. They allow the expert to show the suspected lab site in its original condition. Photographs show the items that were disposed of or otherwise unavailable for presentation at trial. They show the original containers from which evidentiary samples were taken. They can be used in court in lieu of the bulky seized items. The jury can easily handle them during testimony and review them during deliberation. They can be written on to emphasize specific

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aspects of the scene the photograph represents. When placed in the proper sequence, they can be used to prompt narrative testimony, thus telling and enhancing the story of the expert's opinion of what occurred at the location.

The use of photographs has a few disadvantages. The small size prevents the jury from seeing what the chemist is talking about during his testimony, unless the photograph is poster size or each juror has a copy of the photograph being discussed. It is hard to demonstrate how two exhibits physically fit together using photographs. Photographs being handled by the jury during testimony will distract from the actual testimony.

Photographs should be a minimum of 8" × 10". This size enables the jury to minimally see the details in the photograph as the expert describes them. Preferably, the significant portion of the photograph should be marked for later jury review.

7.4.5.2 Slides

Slides have many of the same advantages as photographs. In addition, slides can be projected onto a screen, presenting a larger picture for the jury to see during the expert's testimony. This allows the expert to point out specific items of interest to be emphasized during his testimony.

Slides also have disadvantages. The room lights may have to be dimmed, thus obscuring the jury's view of the expert. If the lights are not sufficiently dimmed, the jury will not be able to see the image. Slides are hard to review in the jury room without a projector. It is hard to demonstrate how two exhibits physically fit together using slides. Photographs can be taped together or placed together on a bulletin board to demonstrate a relationship. Slides cannot. They cannot be written on to stress a point, indicate an exhibit number, or indicate a relationship to another exhibit.

7.4.5.3 Flip Charts and Overheads

Flip charts and overhead projectors can be some of the most versatile of visual aid media available to the expert. They can be made in advance using simple, easy to understand lists or diagrams. The jury can easily see them during testimony. They can show interrelationships between exhibits. One chart can be used for both general and specific explanations. They can be written on during testimony to stress points. Lightly penciled marks can be placed on them to refresh the expert's memory, as long as all of the marks are written over during the testimony in such a way as to be completely visible to the court. They can be taken into the jury room during deliberation.

7.4.5.4 Power Point Presentation

Computers have provided experts with a powerful tool that can be used in making courtroom presentations. Software packages like PowerPoint™ and

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Presentation™ provide the expert the ability to present a choreographed multimedia show to the jury. Photographs can be inserted. Color can be effectively utilized. Diagrams can be presented and altered as the testimony progresses. Bells and whistles can literally be added if the expert thinks it will assist in making his point.

The downside of this technology is that it is currently expensive to implement. Many courtrooms may not possess the projectors necessary to present the show to the jury. Unless the expert brings his own computer and projector, his presentation may have to stay on the computer disc on which it was saved.

7.4.5.5 Evidence Exhibits

Using the actual items seized from a clandestine drug lab during the chemist's testimony is impressive. The jury can see the actual items that were seized from the lab. They can see how the items can be connected to make the reaction apparatus that was described to them.

However, there are problems with using the actual items. The actual items are generally disposed of at the scene and are unavailable for court. If the items were not disposed of, they may still pose a potential chemical hazard, again making them unavailable. The sizes of the items may prohibit their use in the courtroom.

7.4.5.6 Combination of Visual Aids

In the actual court presentation, the expert will use some combination of visual aids. The expert should determine which types of visual aids he prefers and devise a basic presentation concerning how and why this particular set of circumstances constitutes a clandestine lab. The presentation should be flexible enough to include or exclude any type of evidence that is available, because each case will have a different set of evidentiary items with which to work.

Once the basic explanation is established, courtroom presentations of clandestine drug lab evidence should become second nature (with a little practice). Facts concerning individual cases and exhibits used will change, but overall principles will remain the same. Working with the attorney he is testifying for, the expert should be able to mold his presentation to fit the facts of the case.

7.4.5.7 Court Exhibits

The expert should not get attached to his visual aids. As part of the trial process, many jurisdictions require that visual aids be placed into evidence as part of the trial record. That is, the visual aid remains with the court until the case is adjudicated, including through the appeal process. The photo-

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graphs, slides, charts, graphs, and computer files used during the expert's presentation all stay in the courtroom once his testimony is completed. They may be returned once the adjudication process has been completed. However, it would be wise for the expert to take it for granted that he will not see them again and make copies for himself beforehand if he so chooses.

7.5 Summary

Courtroom presentation of forensic evidence is probably the most neglected part of the expert's job. Nowhere is it more important than in the presentation of a clandestine drug lab, for the case often hinges on the expert's opinion. If the expert makes a poor presentation, his information may be lost, and the trier of the fact will not consider it during its deliberation.

The expert's education of the attorney is essential to the successful presentation of a clandestine drug lab case. The attorney must know what made this particular situation a clandestine drug lab so that proper questions are asked. Pretrial meetings are essential for the expert and attorney to devise a script to present all of the information to the court without any surprise questions to the expert or surprise answers to the attorney.

The proper use of visual aids will make an expert's presentation more effective, because the jury will retain five times as much of what they see as opposed to what they hear. The effective presentation of forensic evidence in court is a skill the expert must develop to the same extent as his analytical technique. If the expert is not proficient in courtroom presentation, the most sophisticated evidence in the world may be ignored.

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8 Explosives Labs

The emphasis in this book was placed on the investigation of clandestine drug labs, with explosives and their manufacture discussed as corollary material. This coincides with the author's experience in investigating hundreds of clandestine lab incidents. In his experience, approximately 1% of the operations he responded to involved the actual manufacturing of explosives. While this may seem to be insignificant in the overall scheme of things, it could prove to be the proverbial tip of the iceberg. Most of these operations involving explosives were detected as a result of a clandestine drug lab investigation or were found by emergency medical personnel when responding to a call for assistance.

The events of September 11, 2001 created a heightened awareness of the potential for terrorist acts in the United States. The terrorist bombings at the Murrah Federal Building in Oklahoma City in 1995 (Figure 8.1) and the initial bombing of the World Trade Center in 1993 involved homemade explosive mixtures. At the other end of the spectrum is the young prankster who deposits small but effective homemade explosive devices into the mailboxes of neighbors or someone randomly selected. Somewhere in the middle rests the events that played out at the Columbine High School in Littleton, CO, where a group of teenagers brought numerous homemade explosive devices to school to assist in their carnage. The size of the target audience may differ, as well as the profile of the terrorist, but the effect is the same.

8.1 Explosives Labs Operators and Manufacturers

The motivating forces driving the operator of an explosive lab usually differ from those driving the drug lab operator. The goals of the clandestine explosive manufacturer vary widely. There is one group who directs the final product to an end user (or victim) of the terrorist act. Another group utilizes

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Figure 8.1 The Murrah Federal Building in Oklahoma City after the bombing in 1995.

hazardous materials in booby traps to protect their drug manufacturing operation from law enforcement and rival drug manufacturers. Finally there is the hobbyist, who manufactures explosives and explosive mixtures without criminal intent but for the entertainment value received when they detonate their destructive mixtures.

An assistant U.S. Attorney who was prosecuting a militia group in Arizona in 1998 summarized the motivating factors of the individuals involved in an explosives manufacturing incident. The individuals were charged with manufacturing dangerous devices with the intent to deposit them at federal and local courthouses and law enforcement offices. When asked what the motivating factors of the last two suspects in the case were, the attorney responded. One is on a mission from God. The other one is just stupid.

As inflammatory as this comment was, it summarized two of the motivating factors for clandestinely making explosives. The first portion of the statement indicates that one individual had an ideology or a statement he wanted to express through a violent act, manufacturing the explosives and the explosive device was the route he chose to take. The other part of the statement is consistent with the hobbyist who does not realize the potential damage that could be done to himself or innocent people.

8.2 Regulation

The manufacture of explosives is not an illegal activity per se in the United States. However, the federal government heavily regulates it. The widespread legitimate use of explosives has led to laws controlling their manufacture and

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regulations governing their use. These regulations were formulated to prevent accidents or eliminate incidents that might jeopardize public safety.

Individual jurisdictions may impose additional restrictions on the manufacture of explosives. These laws are dictated with public health and safety in mind. Lack of stiff criminal penalties provides no incentive to initiate an investigation into the clandestine manufacture of explosives unless it is in conjunction with a major felony.

While the manufacturing of explosives may not be illegal, what the end product is used for may be. For example, combining an oxidizer, a fuel, and a sensitizing agent of flash powder may not be illegal in itself. However, when the components are placed into a sealed pipe, the combination becomes a destructive device (a bomb), which is considered a deadly weapon. Coupling the destructive device with the components necessary to create a booby trap increases the intensity of the criminal act. Rigging the booby trap to function further demonstrates the premeditation.

The manufacturing of explosives can be used to demonstrate intent to commit other illegal activities. It can also be used as an aggravating factor to increase the seriousness of a criminal act. For example, the premeditated act of manufacturing an explosive, i.e., using it to construct a destructive device, that is to be used as a booby trap could be used as an aggravating circumstance during trial. These premeditated acts could be construed as assault with a deadly weapon, which would be further aggravated if law enforcement personnel encountered the device.

Fireworks, on the other hand, have a different distinction. Many of their components are classified as explosives in the broad sense by the U.S. government. However, many local jurisdictions classified them as contraband substances, thus making their manufacture and subsequent possession illegal. Such local jurisdictions feel that the general public does not have a legitimate need to possess fireworks. As with drugs, exceptions are well defined. Properly licensed commercial operations can manufacture, possess, and use fireworks under well-defined circumstances. What would the Fourth of July be without a fireworks display? However, in many jurisdictions, the general public is criminally prohibited from all such activities, except when acting as spectators.

The three categories of materials that need to be considered with regard to such clandestine labs are explosives, fireworks, and pyrotechnics. The differences in each of their definitions determine whether their possession or manufacture is illegal or simply regulated. Properly configured, the components of any one of these groups can be incorporated into a destructive device. This act is illegal in all jurisdictions but for different reasons.

The U.S. government defines explosives, fireworks, and pyrotechnic compositions in Title 27, Code of Federal Regulations Section 55.11 (27 CFR 55.11). Explosives are defined as any chemical compound, mixture, or device,

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with a primary or common purpose that is to function by explosion. The term includes but is not limited to dynamite and other high explosives, black powder, propellant powder, initiating explosives, detonators, safety fuses, squibs, detonating cord, igniter cord, and igniters. The list of explosive materials is contained in 27 CFR 55.23 and in Appendix O of this book. Fireworks are defined as any composition or device designed to produce a visual or an audible effect by combustion, deflagration, or detonation, and which meet the definition of "consumer fireworks" or "display fireworks" as defined by this section. Finally, pyrotechnic compositions are defined as chemical mixtures that, upon burning and without explosion, produce visible, brilliant displays, bright lights, or sounds.

By contrast, state and local jurisdictions may address explosives and fireworks under different sections of their legal code. The broad brush that declares the manufacture of drugs illegal on all levels is not generally applicable when it comes to explosives and fireworks. For example, in Arizona, the possession of explosives is addressed in Title 13 (Criminal Code), and the possession and manufacture of fireworks is addressed in Title 36 (Public Health and Safety). Therefore, it is imperative that all federal, state, and local laws be considered during the investigation of explosive manufacturing situations.

8.3 Scene Processing Procedures

The criminal status of the manufacturing of explosives is not an issue when it comes to the procedures used to process the scene of a clandestine manufacturing operation. The procedures are the same as with any clandestine lab, the only difference being that the safety issue becomes much more apparent. Personnel processing the scene must keep in the forefront of their mind the fact that the sole purpose of the operation is to produce a substance that, by definition, is a hazardous material (an explosive). The three general hazards associated with clandestine labs listed in Chapter 2 increase the complication of the processing process at least one order of magnitude.

The operators have little chemical training. They do not understand the hazardous potentials of the chemicals with which they are working. In many instances, the chemicals involved in these operations are on the extreme end of the hazard scale. The acids are some of the most corrosive and possess oxidizing characteristics. The oxidizers are extremely reactive, some to the point of becoming shock or friction sensitive when combined with the right or wrong component.

The lack of understanding of proper laboratory techniques increases this general hazard. Quality control is significant in the world of the clandestine chemist, and certainly not where it should be, in an arena where the purity

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of the final product is critical. Impurities lead to an increased or decreased sensitivity of the compound or mixture, which in turn, leads to unpredictability in the explosive characteristics. The simple fact that the pH is too high or too low may change a relatively stable explosive into one that detonates with the slightest provocation.

As in drug labs, the operator's lack of chemical knowledge leads to the improper storage of chemicals. Ethers exposed to the atmosphere for extended periods of time form explosive peroxides. The accidental explosive potential of picric acid increases dramatically, when it is allowed to dry completely or is stored in a container with a metallic lid.

Finding an unlabeled container is the scariest situation of all. The operator may or may not know what it contains. Accidental detonation of unlabeled containers or unknown substances leads to the detection of many explosive manufacturing operations. It cannot be stressed too often that extreme caution should always be exercised when handling these containers, because the simple act of moving them may cause them to explode.

Curious juveniles operate many clandestine explosive labs. As in clandestine drug labs, their source of information is underground literature or the Internet. The reliability of these recipes is suspect at best. For example, many of the recipes encountered on the Internet have been known to be missing one or more of the steps in the manufacturing process. The closet explosive chemist does not have the technical background to detect missing or additional steps in a chemical reaction. This lack of technical knowledge can lead to the production of a final product that does not work or that is extremely sensitive and explodes with the slightest provocation.

The makeshift nature of clandestine explosive operations increases the potential for disaster. Most reactions are performed under less than ideal conditions using equipment not intended for explosives manufacturing. Sparks, friction, or incompatibility between the chemicals and the reaction vessels can potentially lead to an accident during the manufacturing process or while the emergency responders are trying to identify and abate the hazards.

The sequence of events used to process a clandestine explosives lab scene is the same as a clandestine drug lab. Preraid planning ensures that all of the resources required to safely process the scene are available. The scenario is discussed and the assignments are handed out at the briefing. A trained entry team secures the location as quickly as possible and reports its observations. The evaluation and abatement team identifies and neutralizes any obvious hazards and provides an additional perspective for the search team. The search team processes the site for physical evidence and prepares the site for the disposal company.

The most significant difference between the processing of a clandestine explosive lab and a clandestine drug lab is the interaction between the eval-

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uation and the search teams. In many instances, these functions are combined. The potential for encountering explosive compounds exists throughout the search phase of the operation simply because explosives are the final product. Therefore, the bomb technicians who generally take a passive role during the search of the scene become active participants, as individual items are examined and evaluated.

The documentation of the scene of a clandestine explosive lab is just as important as the documentation necessary for a clandestine drug lab. Even if the manufacturing of explosives is not illegal, the activities that are associated with it may be a serious felony. The manufacturing operation may be construed as the overt act in a homicide or terrorism conspiracy case. Therefore, proper documentation of the scene is essential.

The disposal component of an explosive manufacturing operation takes on a different light. The explosive nature of the end products and many of the precursor chemicals used in the manufacturing process limits disposal options. Many commercial chemical disposal companies will not take explosives or chemicals with explosive potential. If they do, the cost of disposal may be prohibitive.

In these incidences, the local bomb squad often has the authority to perform the disposal operation. This squad has the expertise to safely dispose of the explosive components, which may be completely consumed through combustion or detonation. Even then, regulations concerning the environmental impact of the act must be considered when utilizing this method of disposal.

8.4 Summary

Explosives were invented to cause damage and destruction. The act of clandestinely manufacturing them is not generally illegal in and of itself. Clandestine production often demonstrates a criminal intent for the explosives' end use and can be directly linked to heinous acts of violence and terrorism. The concept is the same, whether it is the mischievous act of blowing up a neighbor's mailbox or the wholesale destruction of an office building, murdering countless innocent occupants. Therefore, if a substance with a legitimate function was manufactured for a criminal purpose, it should be treated with the same fervor.

The concepts associated with the processing of a clandestine explosives lab scene are the same as those used in clandestine drug labs. The hazardous nature of the end product demands extra vigilance when it comes to adhering to safety protocols. The dangerous nature of the end product and the operating conditions used to produce it dramatically increase the potential for disaster through an accidental explosion.

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9 Practical Applications and Examples

Every seminar or workshop instructor presents scenarios and practical applications of the information presented. Usually it is a compilation of events in which the presenter applied the techniques under discussion. In essence, the discussion is a series of the presenter's "war stories." In keeping with that tradition, this is the book's "war stories" section. Contained in this chapter is a compilation of scenarios that are used to demonstrate the various points that were brought up in the previous chapters. All of the events actually happened. However, some of the specific details were changed to emphasize the points being discussed.

Reading through the examples may elicit a variety of responses and questions. Some examples may elicit a response like: "That was stupid. Why did he do it that way?" Another instance may elicit an "I did not realize that would happen" response. Hopefully, the absurdity involved in some of the situations will be demonstrated, as they are reviewed in an objective manner. More importantly, the ramifications of the action or inaction on the part of the forensic investigator in the case at hand will be understood.

Other portions of this chapter are technically oriented. The applications will provide insight into how certain examinations are performed and the results of different analytical approaches will be compared and contrasted. In some instances, a step-by-step description of a process will be provided to detail the reasoning behind each of the steps. Understanding the reasoning behind a process assists in incorporating it into specific situations.

Practical Example 1: Extraction Labs

Extraction labs are set up to remove raw materials from a mixture. This is accomplished by using the desired component's physical and chemical prop-

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Figure 9.1 Empty over-the-counter cold medication containers.

erties to separate it from the mixture. No chemical change in the raw material occurs during the process. The process, in itself, may not be illegal. However, being able to recognize when the process is being used for illicit purposes provides the expert the ability to fill in missing puzzle pieces.

Clandestine lab operators commonly use over-the-counter medications containing the precursor chemicals needed for the production of amphetamine or methamphetamine. They grind them into a powder, and placed them into a jar. A solvent is added to the powder, and the chemicals of interest are dissolved into the liquid, "extracting" them from the insoluble inert components of the tablet. The liquid is decanted into a glass pie pan that is placed on an electric hot plate to evaporate the solvent. The residue contains a relatively pure form of the desired precursor chemical.

Figures 9.1 and 9.2 show examples of an extraction lab at which three extraction processes are taking place. First, the precursor was chemically removed from the medication with the solvent. Second, the liquid was physically separated from the solids. Finally, the solvent was physically removed from the precursor through evaporation.

Possession of over-the-counter medications is not illegal. The extraction of the precursor chemical components of the tablets may not be illegal. However, the combination of the quantity of tablets and the extraction process may be used to establish intent to conduct an illegal activity.

Practical Example 2: Extraction Labs

Numerous vials of a veterinarian drug preparation containing ketamine were found at a clandestine lab located in a bungalow in a luxury resort. The operators were removing the solution containing the drug from the injection

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Figure 9.2 Precursor chemical extraction laboratory.

vials and evaporating the liquid. The resulting powder contained ketamine hydrochloride, which was a controlled substance under local statutes. The operators were convicted of manufacturing a controlled substance in addition to possessing a controlled substance. They "extracted" the drug from the original mixture, which was included in the definition of manufacturing under the local statute.

Practical Example 3: Conversion Labs

Conversion labs are one of the most commonly encountered clandestine labs. In a conversion lab, a raw material is changed into the desired product. This process involves making minor structural changes within the molecule of the compound or of the chemical's salt form. Functional groups may be added or removed from the molecule, somewhat like building with Tinkertoy® pieces. The drug of interest can also be converted from its salt form to the freebase form or from the freebase form to the salt form.

Simply changing the pH of a water solution containing cocaine hydrochloride produces "Crack" cocaine. Changing the pH with a basic reagent chemical removes the hydrochloride component from the cocaine molecule. This changes the cocaine's salt form, making it insoluble in water. It also lowers its boiling point, allowing it to be smoked.

This type of conversion lab can be encountered in a variety of situations. An individual can carry all of the components in his pocket. A vial of water and a small amount of baking soda are all that is necessary to convert cocaine hydrochloride into Crack. Larger-scale operations are slightly less mobile, but the same ingredients are utilized and can be encountered at or in the vicinity of the distribution point.

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Figure 9.3 Phencyclidine precursor and reagent chemical.

Practical Example 4: Conversion Labs

The Grignard reagent is a very reactive compound that is commonly used in chemical synthesis. The reaction between bromobenzene and magnesium produces phenylmagnesium bromide, a very reactive Grignard reagent. This compound is an essential intermediate component used in the synthesis of phencyclidine and its analogs. This reaction is self-driven and can be accomplished in a plastic bucket.

In response to information provided by a local street person, investigators found chemicals concealed behind trash cans in an alley (Figure 9.3). A plastic bucket containing a liquid with a strong ether odor with gray metallic particles at the bottom was found a short distance away (Figure 9.4). Laboratory analysis determined that the liquid contained bromobenzene, and the metallic particles were identified as magnesium turnings. The expert concluded that the Grignard reagent located in the plastic bucket was to be added to the phenylcyclohexylcarbonitrile (PCC) that was located in the vicinity to synthesize phencyclidine (PCP).

Practical Example 5: Conversion Labs

Ephedrine or pseudoephedrine is converted into methamphetamine using a simple reduction reaction. In the chemical reaction, a hydrogen atom is substituted for a hydroxyl group (-OH) to produce methamphetamine. The

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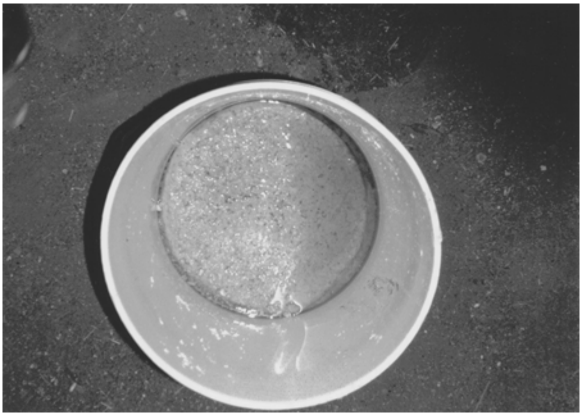

Figure 9.4 Phencyclidine reaction mixture.

skeleton of the molecule is intact. However, the physiological effect of the drug on the body is dramatically different.

Traditionally, the conversion of ephedrine into methamphetamine was accomplished by using a hydrogenator or a reflux apparatus. However, the ingenuity of the clandestine lab operator created a situation in which this conversion can be accomplished using ordinary kitchen utensils. Understanding the basic principles involved in the conversion of the precursor chemical into the final product will allow the investigator to recognize ordinary items that have been adapted for use in a clandestine manufacturing operation.

A clandestine lab chemist was asked if a pressure cooker could be used to manufacture methamphetamine using the ephedrine/HI reduction process. Using the "cooking soup" analogy, the chemist advised the investigator that the process described by the informant was viable and would produce the desired result. A pressure cooker and mason jars were found during the subsequent search of the location. The operator used the mason jars as reaction vessel and condenser. The pressure cooker was used as the heating mantle. A condenser was not required, because the closed mason jars inside the pressure cooker produced a closed system that contained the fumes that would normally have been condensed or vented away from the reaction. Figures 9.5 and 9.6 show examples of a pressure cooker that was used to produce methamphetamine in this fashion.

Practical Example 6: Synthesis and Extraction

The synthesis process is a chemical reaction or series of chemical reactions in which molecules or parts of molecules are combined to create a new

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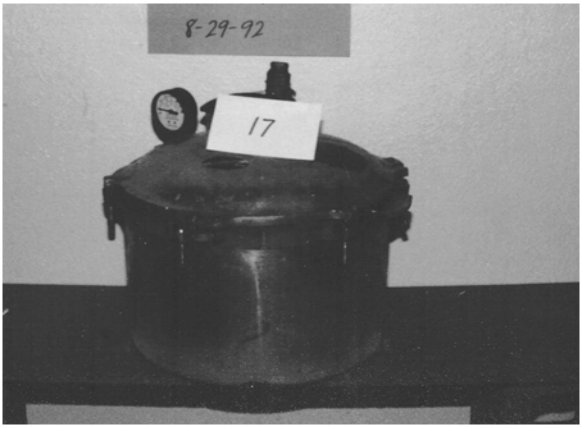

Figure 9.5 Pressure cooker reaction vessel.

Figure 9.6 Interior of pressure cooker.

molecule. This process can be equated to a chemical-type Erector® set. It differs from the conversion process in that the skeleton of the resulting molecule is a sum of the molecules or significant parts of the molecules involved in the reaction. Lysergic acid diethylamide (LSD), phencyclidine (phenylcyclohexyl piperidine, PCP), phenylacetone (P2P), and certain methamphetamine reactions are examples of drugs produced using the synthesis process.

Phencyclidine is produced in a multistep reaction. During the process, bromobenzene, cyclohexanone, and piperidine are chemically combined in a two-step process. The resulting molecule has the combined chemical skeletons of all three precursor chemicals.

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Figure 9.7 Bucket chemistry example of PCP lab.

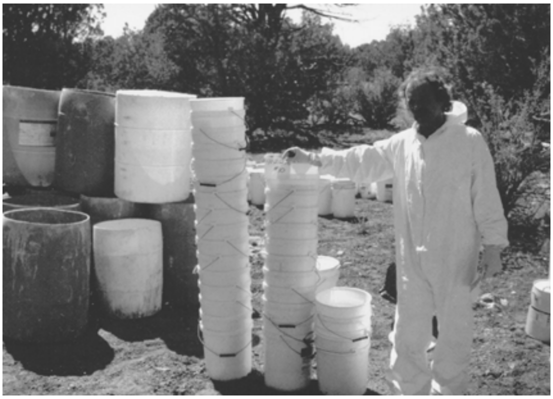

Figure 9.8 Large-scale PCP lab.

The manufacturing of PCP is so simple that it is commonly described as "bucket chemistry." The equipment required for this synthesis reaction is shown in Figures 9.7 and 9.8. The images demonstrate that the required equipment can be common and ordinary and does not have to be sophisticated or exotic. In this example, it was estimated that over 100 pounds of PCP was produced in a rural operation, in which all of the chemical reactions were conducted in 5 gal plastic paint buckets.

Practical Example 7: Distillation

A distillation apparatus can be used to simultaneously synthesize and extract phenylacetone. The precursor and reagent chemicals are combined in the

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reaction flask and heated to a rolling boil. As the mixture boils, the chemicals react, producing phenylacetone and its associated reaction by-products. The by-products, with a boiling point lower than phenylacetone evaporate, are separated, collected, and discarded through the distillation process. Once the boiling point of the reaction mixture reaches that of the phenylacetone, the separated liquid is saved. The heat is removed from the reaction when the mixture's temperature rises above the boiling point of phenylacetone.

Practical Example 8: Distillation

Figure 9.9 illustrates an example of a situation in which a little knowledge can be dangerous. The operator in this situation understood the basic concept concerning distillation. However, he arranged a distillation apparatus such that the reception flask was above the boiling flask. This arrangement resulted in nothing more than a modified reflux apparatus. Gravity returned the liquid from the condensing vapors to the boiling flask instead of separating it into the reception flask. The operator had no idea why the apparatus was not working.

Practical Example 9: Distillation

The operator in the situation illustrated in Figure 9.10 constructed a vacuum distillation apparatus to purify phenylacetone. The boiling container was constructed from an 8" steel pipe with metal plates bolted onto the top and bottom. A kitchen hot plate was used to apply heat to the boiling container. The condenser was constructed of two sizes of copper tubing. Cool water was circulated through the makeshift condenser using a submersible water pump and a trash can containing ice water. The reception flask was a vacuum flask connected to a beer keg that was connected to a commercial vacuum pump. This apparatus functioned very well.

Practical Example 10: Extraction and Separation

During the final stages of a methamphetamine synthesis or conversion reaction, the reaction mixture is cooled to room temperature. The pH of the solution is changed, and an organic solvent is added to the mixture. The methamphetamine dissolves in the organic layer. The liquid combination is placed into a separate container, and the organic layer is isolated and separated through the use of a traditional separatory funnel (Figure 9.11). The liquid combination is placed into the funnel and allowed to form two distinct

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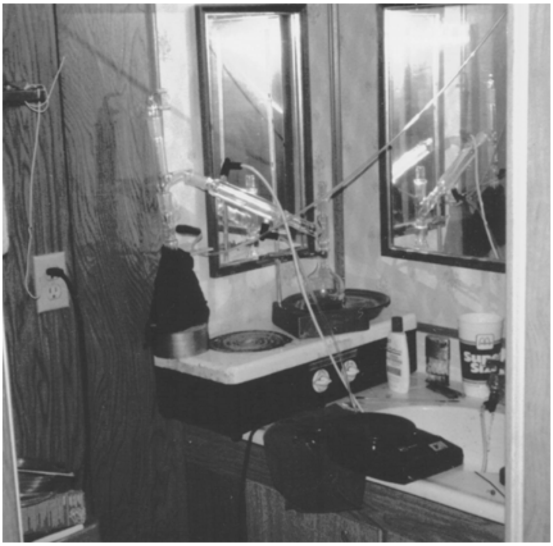

Figure 9.9 Inverted condensing column on a distillation apparatus.

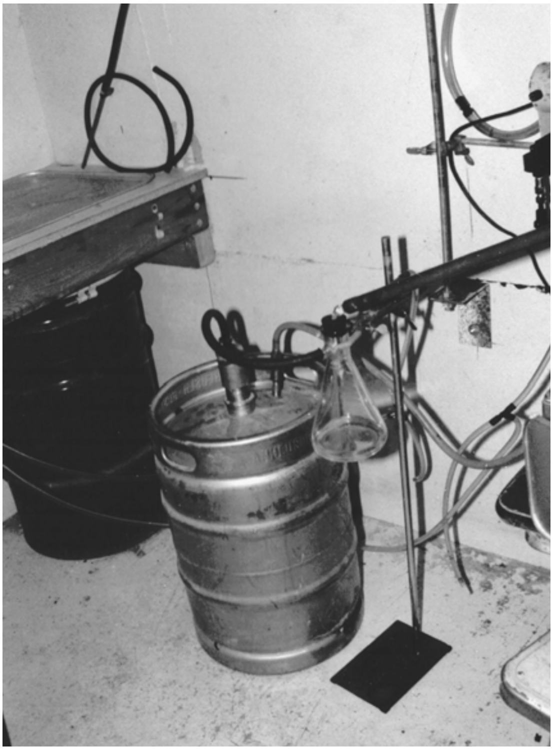

Figure 9.10 Homemade vacuum distillation apparatus.

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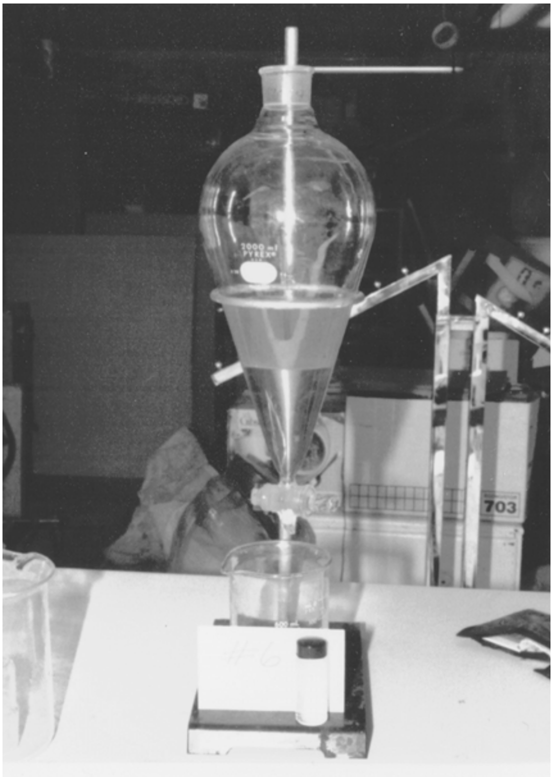

Figure 9.11 Conventional separatory funnel containing a two-phase liquid.

layers. The valve is opened, and the lower liquid is allowed to drain into a receptacle. The valve is then closed when the line defining the separation between the top and bottom layers reaches the valve.

Sport bottles (Figure 9.12) can be used in a similar manner. The combination of liquids is placed into the bottle, the cap is put into place, the liquids are allowed to separate, and the bottle is then inverted. A vacuum is created in the air space above the liquids, keeping the liquids from pouring out of the bottle. The lower liquid is removed by gently squeezing the bottle, forcing the liquid out of the restricting valve at the opening.

Practical Example 11: Filtration

Hydrogen chloride gas is added to the organic solution from Practical Example 10. The freebase drug and the gas react to produce the hydrochloride salt form of the drug, which is insoluble in the organic liquid. The solid–liquid mixture is poured into a Buchner funnel attached to a vacuum flask (Figures

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Figure 9.12 Plastic soda bottle used as a separatory funnel.

9.13 and 9.14). The liquid is drawn into the vacuum flask when the vacuum is applied to the system, leaving the solid in the Buchner funnel. Acetone can be used to remove the reaction by-products from the solid. It is poured over the solid and drawn by vacuum into the flask. As the by-products are removed, the solid turns white.

In this example, the drug was chemically extracted from the liquid by changing its salt from. The solid was then physically extracted from the liquid through vacuum filtration. Finally, the reaction by-products were chemically and physically extracted from the drug in a tandem operation. The acetone chemically extracted the by-products, and was simultaneously physically extracted from the drug, while being filtering under vacuum.

Practical Example 12: Mechanical Explosions

A mechanical explosion occurs when the structural integrity of a container is compromised as the result of excess pressure inside the container. This

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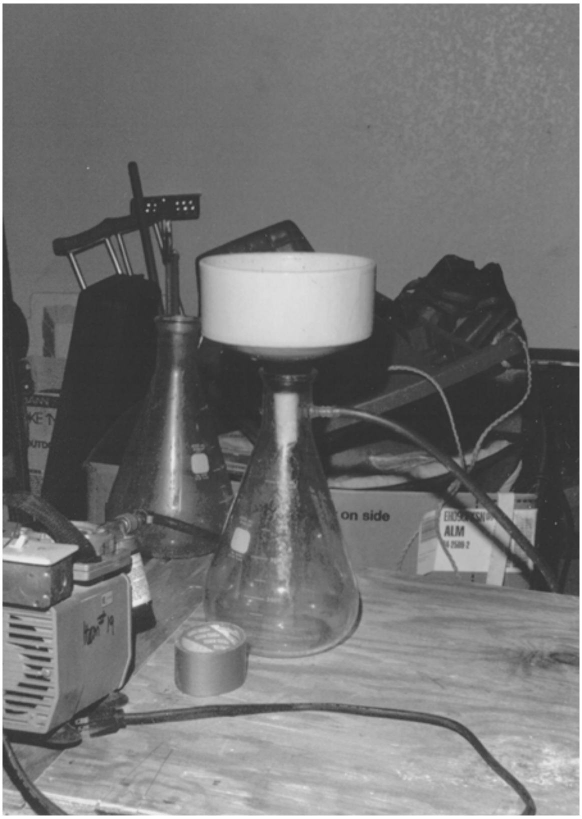

Figure 9.13 Vacuum filtration.

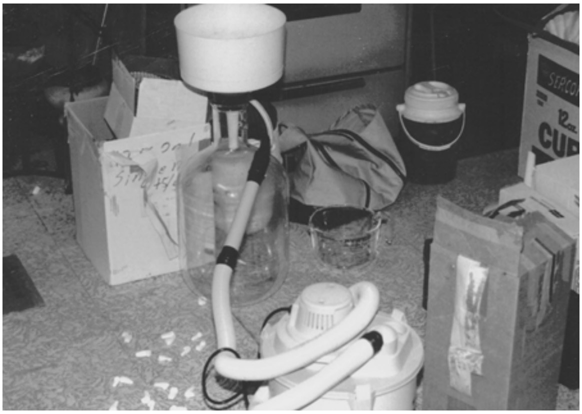

Figure 9.14 Vacuum filtration with shop vac as vacuum source.

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situation can occur by design, as in the case of a pipe bomb. However, when associated with a clandestine lab, it is more often a result of equipment modification performed by the operator. The following examples are results of operator equipment modifications.

The operator attempted to vent the fumes emanating from the top of a reflux condenser into a makeshift filtering device. As a result, the opening of the condensing column became obstructed. The strength of the boiling flask was compromised by stress cracks that were the result of improper handling. The pressure would build to a point at which one of three things could happen. First, the pressure could clear the obstruction at the top of the condensing column. The excess pressure would be vented into the filtering device. This may or may not have an adverse effect, depending upon the construction of the filter.

Second, the connection between the boiling flask and the condensing column could release. The pressure in the system would then propel the condenser like a missile into the ceiling. The contents of the reaction flask would spew from the reaction flask opening like a fountain, bathing everything in the area with boiling hazardous chemicals.

The other option is that the reaction flask could lose its structural integrity. It would shatter, and the pressure from the system would propel the broken pieces of glass like shrapnel from a grenade and coat the immediate area with the boiling reaction mixture. This option contains the dual hazard of chemical exposure and of being impaled by reaction flask fragments.

Practical Example 13: Mechanical Explosions

A clandestine lab operator repeatedly used a kitchen pressure cooker as a reaction vessel. Over time, the pressure relief valve corroded, so he soldered the opening closed. This particular operator placed the reaction mixture directly into the altered pressure cooker, placed it on the electric kitchen stove, and turned on the stove. The acids in the reaction mixture weakened the metal during repeated use of the pressure cooker. The pressure from the boiling reaction mixture reached the point at which the pressure cooker lost its structural integrity and exploded. The metal lips on the pot portion, which held the lid on, sheered off. The pressure propelled the lid into the stove vent, spewing hot reaction mixture over the immediate area. Fortunately, no one was in the vicinity of the kitchen at the time of the explosion.

Practical Example 14: Vapor Explosions

The operator of a large-scale gamma hydroxy butaric acid (GHB) operation constructed a drying room in which to evaporate the residual acetone from

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Figure 9.15 Vapor explosion fire damage.

Figure 9.16 Vapor explosion blast damage.

his final product. During the drying process, the concentration of the acetone vapors inside the drying room reached the explosive range. A spark was generated inside the drying room when the operator turned on an interior light. This resulted in a vapor explosion with effects that are demonstrated in Figures 9.15 and 9.16.

Practical Example 15: Compressed Gas Hazards

Compressed gases are utilized in a variety of situations in the clandestine manufacture of controlled substances, and they pose multiple hazards. First,

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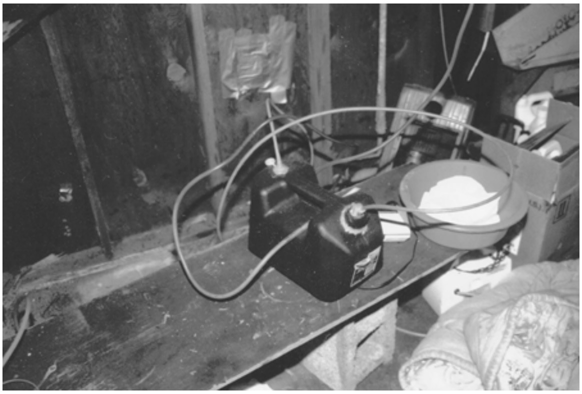

Figure 9.17 Hydrogen chloride gas generator.

the chemical within the container may have a hazardous component. Second, the container may be unstable and pose a physical hazard to anyone attempting to handle it. Third, because of the second condition, there may be no safe way to determine the status of the first. Simply put, the investigator does not know what is in the container, and the container's condition may be too hazardous to determine what is inside. The following examples are used to provide some insight as to the hazardous potential of compressed gas containers encountered in clandestine labs.

Hydrogen chloride gas ($HCl_{(g)}$) is used to convert freebase drugs into the hydrochloride salt form. One method bubbles commercially available $HCl_{(g)}$ into a mixture of extraction solvent and freebase drug. The freebase drug reacts with the $HCl_{(g)}$, creating a solid that precipitates out of solution. Clandestine lab operators commonly generate their own $HCl_{(g)}$ using household chemicals. They place the chemical mixture into containers, such as compressed gas cylinders, plastic gas containers, or other containers that can be sealed and in which the pressurized gas is vented in some manner. The result is a pressurized container of $HCl_{(g)}$.

The metal that compressed gas cylinders are constructed of is incompatible with the $HCl_{(g)}$. The $HCl_{(g)}$ corrodes the brass valves or reacts with the metal of the container. The corroded valve can break during use, or the container may eventually lose its structural integrity. Both situations lead to a discharge of pressurized $HCl_{(g)}$.

The other situations are not much safer. The containers used by the operators may be resistant to the corrosive nature of the chemicals involved, but they are not designed to withstand significant pressures. As a result, $HCl_{(g)}$ is continually placed into the atmosphere until the chemical reaction between the ingredients has finished.

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Practical Example 16: Compressed Gas Hazards

Clandestine lab operators utilizing the Birch reduction (commonly referred to as the "Nazi" method) obtain the liquid ammonia required from agricultural areas that use it as a fertilizer. The operators use the propane tank from a gas barbecue to transport and store the stolen ammonia. Over time, the ammonia corrodes the valve on the tank to the point at which the valve does not function. It may break when operated, resulting in the release of pressurized ammonia into the atmosphere.

Practical Example 17: Compressed Gas Hazards

Under pressure, ephedrine can be reduced into methamphetamine in the presence of a catalyst, acid, and hydrogen. Clandestine lab operators have designed an apparatus that uses a 2 l plastic soda bottle that will facilitate the hydrogenation process. Under normal conditions, plastic soda bottles can maintain pressures in excess of 500 lb/in^2. However, the conditions in a hydrogenation reaction expose the bottle to temperatures and chemicals the container was not designed for. Constant increases and decreases of pressure during the hydrogenation process, coupled with the heat generated by the chemical reaction, compromise the structural integrity of the bottle to the point where any shock will cause the plastic skin of the bottle to rupture and peel open like an over-ripe watermelon, releasing pressurized hydrogen and corrosive chemicals.

Practical Example 18: Initial Crime Scene Evaluation

Everything done at a crime scene potentially has evidentiary value. Every action and word has the potential of finding its way into the court. Nowhere was this more apparent than in the O. J. Simpson case, in which the investigators spent hours on the witness stand explaining comments and personal opinions that were expressed during the crime scene investigation. Provided in this section are examples of how actions taken at a clandestine lab scene may have ramifications in later stages of the investigation.

A clandestine lab scene chemist and the lead investigator began processing the scene of a suspected clandestine drug lab after the scene had been secured and the hazards were abated. The lead investigator, who only had a minimal knowledge of clandestine manufacturing techniques, immediately began videotaping the scene and providing audio descriptions of how each chemical and piece of equipment would be used to manufacture methamphetamine.

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The scene chemist, who had 10 years of experience in clandestine lab processing, was performing a preliminary walk through at the same time. Within minutes, the scene chemist realized that the operator was manufacturing explosives, not drugs. He stopped the processing and evacuated the lab area to revise the processing plan. When reading the physical evidence, the scene chemist realized the operator was manufacturing nitroglycerine, not methamphetamine. This piece of knowledge radically affected how the balance of the scene was processed.

Practical Example 19: Training and Experience

A forensic chemist who was not trained in clandestine lab manufacturing methods arrived at the scene of a suspected methamphetamine lab. Without evaluating the combination of chemicals and equipment that was present at the scene, he pronounced that the operation was manufacturing methamphetamine. He took a minimal amount of samples and left the scene. Using the scene chemist's opinion, without corroborating laboratory analysis, the lead investigator charged the operator with manufacturing methamphetamine.

A clandestine lab chemist subsequently evaluated the physical evidence from the scene. His laboratory examinations of the evidentiary samples, evaluation of the scene's photographs, and review of the chemicals revealed that the operator was manufacturing diethyltriptamine, a hallucinogen, not methamphetamine. The haste actions of the scene chemist and the lead investigator led to the dismissal of the charges against the operator.

Practical Example 20: Training and Experience

In contrast to Practical Example 19, demonstrated in this practical example are the positive effects of having qualified personnel read the physical evidence and adjust the scene processing protocols as more information is discovered.

A clandestine lab response team properly secured and abated the hazards at a site run by an educated commercial operator who had a history of manufacturing gamma hydroxybutaric acid (GHB). The operation under investigation was clean and nonoperational. During their evaluation of the chemicals and the operator's notes, the scene chemists determined that the operator was experimenting with the manufacture of meperidine and fentanyl analogs. These compounds have been linked to Parkinson's disease. At this point, the hazard potential dramatically changed, as did the approach to the way the scene would be processed. The lab area was evacuated, and the scene-processing plan was revised.

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Practical Example 21: Sampling

The following is an example of the ramifications of improperly sampling a clandestine lab scene. The scene chemist in this situation did not possess training in clandestine lab scene processing. He did an admirable job of photographing the scene, which allowed the clandestine lab chemist to render some opinions after the fact.

Three containers located at the scene of this clandestine drug lab were identified as containing an acid. One container was a plastic gasoline container containing a clear acidic liquid that produced white fumes when the container was opened. The two other containers were commercial 500 ml clear glass bottles with black caps. The labels on the bottles had been removed. The bottles contained a clear acid liquid with a yellow tint. Photographs were taken of each of the containers. Only the red plastic container was sampled. Subsequent laboratory analysis of the sample revealed that the contents were consistent with hydrochloric acid.

Below is an excerpt of the analytical chemist's testimony. The defense contended that lack of the presence of hydriodic acid precluded the operator from manufacturing the controlled substance that the state contended. Cross-examination of the analytical chemist charged with analysis of the evidence proceeded as follows:

Defense Attorney: You stated exhibit 12 contained hydrochloric acid?

Chemist: Yes sir.

Defense Attorney: So there was no hydriodic acid found at the scene?

Chemist: No Sir, I cannot say that.

Defense Attorney: But your report states that you found hydrochloric acid, not HI. How can you say that there was hydriodic acid present?

Chemist: The items in exhibit 24 and exhibit 31 were not sampled. The packaging and the color of the liquid are consistent with hydriodic acid.

Defense Attorney: But your report states that hydrochloric acid was the only acid identified.

Chemist: That is correct. However, the items in exhibit 24 and 31 were not sampled so I could not analyze the contents. Without laboratory analysis I cannot comment on the contents.

Defense Attorney: So you are saying you did not find any hydriodic acid?

Chemist: What I am saying is that I cannot say that there was no hydriodic acid at the scene. The packaging and color of the liquid of items 24 and 31 is consistent with commercially packaged hydriodic acid.

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This whole exchange could have been avoided if the items at the scene were sampled properly. This would have provided the analytical chemist the opportunity to identify the contents of each container.

Practical Application 1: Bottle Volume Estimates

In some jurisdictions, the penalty associated with crime is related to the amount of controlled substance seized. Many statutes use the phrase, "at time of seizure," as the benchmark time. In simple possession or possession for sale cases, this value is easily determined by weighing the substance on a calibrated balance during the laboratory examination of the exhibit. This can be problematic in clandestine lab investigations, because a majority of the evidence is disposed of due to its hazardous nature. The 2 fluid oz sample that is retained for laboratory examination may hardly be representative of the volume of substance that was seized. Without documentation to support a larger volume/weight, the only value the court can rely on to establish a sentence would be the weight/volume of the representative sample that was submitted for laboratory examination. If the dimensions of the containers and their contents are documented, simple geometry can be used to establish the original volume of the substance. Thus, this will give the court the "at the time of seizure" value to use to establish the sentence.

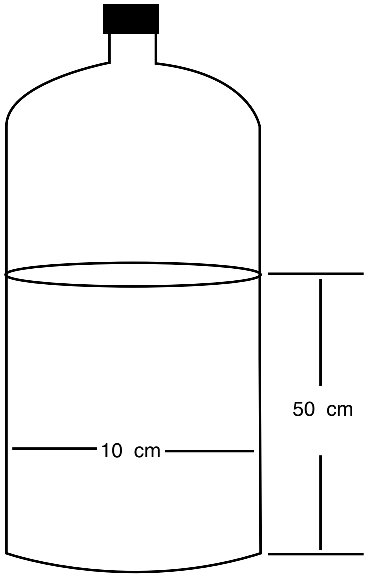

Figure 9.18 Bottle containing liquid.

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The volume of the contents of a clear glass bottle must be determined. The cylindrical-shaped bottle has a diameter of 10 cm. The height of the liquid in the bottle is 50 cm. The volume of the liquid can be calculated using the simplified cylinder volume equation (Figure 4.1) as follows:

$$\text{Volume}_{\text{cylinder}} = 0.78 * \text{diameter} * \text{diameter} * \text{height}$$
$$= 0.78 * 10 \text{ cm} * 10 \text{ cm} * 50 \text{ cm} = 3900 \text{ ml} = 4.1 \text{ qt}$$

Practical Application 2: Flask Volume Estimates

The volume of a clear yellow liquid in an Erlenmyer flask is needed. The Erlenmyer flask has a conical shape, is 30 cm tall, and has a base diameter of 20 cm. The height of the liquid in the flask is 10 cm, and the diameter of the flask at the top of the liquid is 10 cm.

Calculating the volume is a three-step process. The total volume of the flask is calculated first. Second, the volume of the air on top of the liquid is calculated. Finally, the air volume is subtracted from the total volume to determine the volume of the liquid in the flask. Using the equation from Figure 4.2, the calculation is as follows:

$$\text{Volume}_{\text{total}} = 0.26 * \text{diameter} * \text{diameter} * \text{height}$$
$$= 0.26 * 20 \text{ cm} * 20 \text{ cm} * 30 \text{ cm} = 3120 \text{ ml}$$

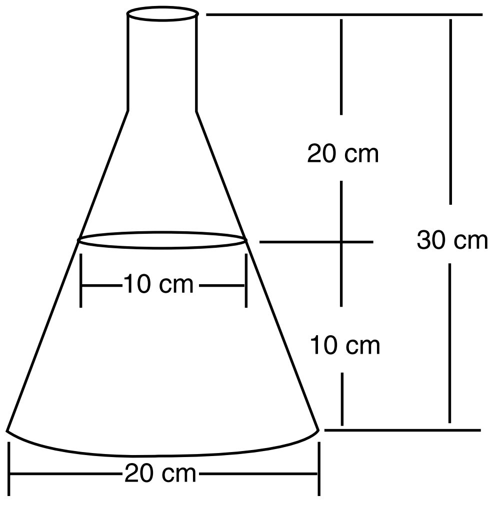

Figure 9.19 Flask containing liquid.

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$$\text{Volume}_{\text{air}} = 0.26 * \text{diameter} * \text{diameter} * \text{height}$$
$$= 0.26 * 10 \text{ cm} * 10 \text{ cm} * 20 \text{ cm} = 520 \text{ ml}$$

$$\text{Volume}_{\text{bottom}} = \text{Volume}_{\text{total}} - \text{Volume}_{\text{air}}$$
$$= 3120 \text{ ml} - 520 \text{ cm} = 2600 \text{ ml} = 2.7 \text{ qt}$$

Practical Application 3: Separatory Funnel Volume Estimates

A separatory funnel is found containing a two-phase liquid. The bottom layer is 5 cm high, and the top layer is 10 cm high. The diameter of the funnel at the point the two liquids meet is 10 cm. The diameter of the funnel at the top of the top liquid is 20 cm. What is the volume of both liquids?

The basic shape of a separatory funnel is that of an upside-down cone. Therefore, the same method that was used to calculate the volumes in the flask is used for this calculation. The total volume in the funnel occupied by liquid is the first thing to be calculated. The next step is to calculate the

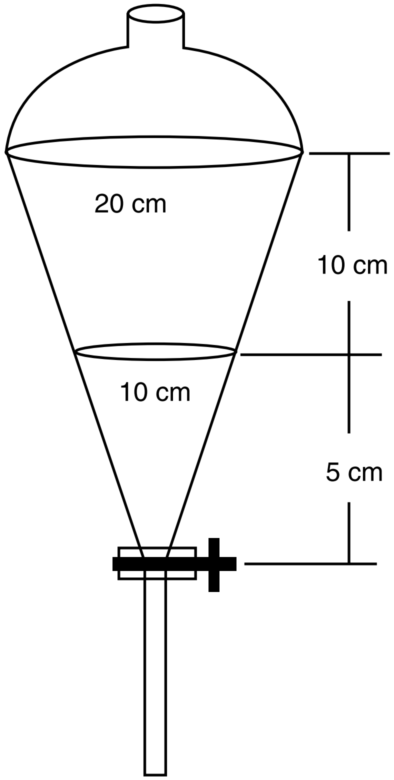

Figure 9.20 Funnel containing liquid.

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volume in the liquid phase closest to the apex of the cone. Finally, the lower layer volume is subtracted from the total volume to establish the volume of the upper layer.

$$\text{Volume}_{\text{total}} = 0.26 * \text{diameter} * \text{diameter} * \text{height}$$
$$= 0.26 * 20\text{ cm} * 20\text{ cm} * (10\text{ cm} + 5\text{ cm}) = 1560\text{ ml} = 1.64\text{ qt}$$

$$\text{Volume}_{\text{bottom}} = 0.26 * \text{diameter} * \text{diameter} * \text{height}$$
$$= 0.26 * 10\text{ cm} * 10\text{ cm} * 5\text{ cm} = 130\text{ ml} = 0.5\text{ cup}$$

$$\text{Volume}_{\text{top}} = \text{Volume}_{\text{total}} - \text{Volume}_{\text{bottom}}$$
$$= 1560\text{ ml} - 130\text{ cm} = 1430\text{ ml} = 1.51\text{ qt}$$

Practical Application 4: Reaction Flask Volume Estimates

The calculations to establish the volume of a sphere that is filled with liquid are more complicated than the simple mathematics used in the previous examples. In July, 1991, the DEA published a table of partial sphere volumes based on reaction flask size and liquid height. This table was reproduced in Appendix P. The diameter of the reaction flask is used to establish its volume. The height of the liquid in the flask is then cross-referenced in the table in Appendix P to obtain the amount of liquid in the reaction flask.

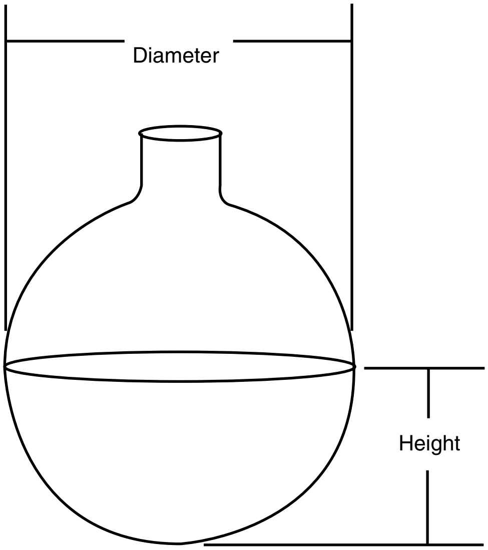

Figure 9.21 Reaction flask containing liquid.

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Practical Example 22: Data Interpretation

The laboratory analysis of samples taken at the scene of a clandestine laboratory can generate a significant amount of information. The analytical methods traditionally utilized by the forensic chemist analyzing the samples can provide the answers to the questions the expert needs to provide an informed opinion concerning the operation under investigation. However, the analytical chemist needs to know what questions should be answered so he can apply the appropriate analytical technique to the samples being analyzed. In this section, a few examples of how the information provided by the analytical chemist is used by the clandestine lab chemist to reach his conclusions are provided.

The inorganic analysis of an off-white crystalline substance from a suspected methamphetamine lab reveals the presence of sodium, phosphorous, iodine, and a relatively small amount of chlorine. The chemist must account for the presence of each of the elements. From the notes seized at the scene and the chemical inventory, it is the chemist's opinion that the operator was probably using the HI/red phosphorous method of reducing ephedrine to methamphetamine. Using this as a basis, the chemist reasons that the iodine originated from the HI, the phosphorous was from the red phosphorous, and the sodium hydroxide used to neutralize the HI contributed the sodium. Finally, the small amount of chlorine is assumed to have originated from the hydrogen chloride salt of the ephedrine that was used as a precursor chemical.

Practical Example 23: Data Interpretation

A red sludge material recovered from a trash search was submitted for laboratory analysis to help develop the probable cause necessary for a search warrant for the location of a suspected clandestine lab. The chemist is asked what method was being used to manufacture the methamphetamine without being supplied a list of chemicals or recipe from the operator. To provide the requisite opinions, the clandestine lab expert combines the information from the organic and inorganic chemical profiles to determine the controlled substance being manufactured and the method of manufacturing.

The organic analysis of the sample revealed the presence of methamphetamine, ephedrine, and a trace of phenylacetone. The ratio of these components indicated the operator was reducing ephedrine to methamphetamine using hydriodic acid. This was consistent with the chemist's expectations from his visual inspection of the sample prior to analysis.

The inorganic analysis revealed the presence of potassium, iodine, phosphorous, and chlorine in relatively equal amounts, with a small amount of

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sulfur present. This inorganic profile presented a quandary. It was not consistent with the information provided by the organic analysis. The unusually high concentration of chlorine and potassium, in combination with the presence of sulfur, were of concern to the clandestine lab chemist.

To rectify the discrepancy between the organic and inorganic results, the clandestine lab chemist had to think "outside the box," utilize his knowledge of organic synthesis, and incorporate alternative methods used by clandestine lab operators in the past. He referred to a method that substitutes a strong acid and a source iodide, usually from an iodine salt, to reduce the ephedrine. Using this information, he proposed that the potassium and iodine were from a potassium iodide salt, the chlorine was from hydrochloric acid, and the phosphorous was from the red phosphorous. The small amount of sulfur was attributed to the sulfate salt form of the ephedrine that was used as a precursor. When the operation was seized, the chemical inventory and the operator's notes confirmed the chemist's proposal.

Practical Example 24: Data Interpretation

All samples should be given the same value and analytically be treated the same. It is easy for the analytical chemist to become complacent and dismiss a sample, because it "looks" a certain way. There is some validity to the initial observations of a seasoned analytic chemist. However, his "gut feeling" should not be substituted for a documentable scientific analysis. The following example demonstrates how misleading gut feelings can be.

An analytic chemist received a sample of a clear liquid seized from a freezer located in a clandestine lab that was operating in a government laboratory. The sample was from a labeled container, and its odor and general appearance were consistent with the label. The chemist was prepared to dismiss the sample as "contents consistent with the label" without analytical data to support his conclusion. However, much to his surprise, he detected a significant amount of methylenedioxyamphetamine (MDA) when he performed a routine gas chromatographic screen on the sample.

Practical Application 5: Dry Extractions

There are a number of different extraction techniques available to the analytic chemist. Each has its place in the toolbox of the analytical chemist. The challenge is for the analytical chemist to choose the one that is most appropriate for the sample under examination. The following practical examples are applications of various extraction techniques used in the analysis of clandestine lab samples.

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Cocaine HCl is generally found in a powder matrix mixed with one or more adulterants or diluents. Many of these compounds can be removed through one or more dry extractions. The following are two techniques that can be used to isolate cocaine HCl without altering its salt form:

Dry Extraction 1
- Physically isolate particles that appear to be pure cocaine HCl with tweezers.
- Analyze the particles using IR, which identifies the cocaine as well as establishes the salt form. The sample can be analyzed via GC/MS if the salt form of the cocaine is not an issue.

Dry Extraction 2
- Dissolve powder sample in chloroform. (Cocaine HCl is soluble in chloroform, and most common diluents and adulterants are not.)
- Separate the chloroform from the powder.
- Evaporate the chloroform.
- Analyze the residue via IR to confirm the presence of cocaine as well as establish the salt form. The sample can be analyzed using GC/MS if the salt form of the cocaine is not an issue.

Dry Extraction 3
- Dry wash the powder sample with ethyl ether. (This removes assorted impurities, i.e., niacinamide and any freebase cocaine that might be present.)
- Dry wash the sample with acetone. (This will remove common diluents, i.e., lidocaine HCl.)
- Analyze the residue via IR to confirm the presence of cocaine as well as establish the salt form. The sample can be analyzed using GC/MS if the salt form of the cocaine is not an issue.

These extractions may or may not remove all of the auxiliary components from the sample, allowing it to be confirmed as cocaine. However, enough of them should be eliminated to establish if a salt form exists and, if so, which one it is.

Practical Application 6: Methamphetamine Extraction

Just as the salt form of cocaine may be significant, the salt form of methamphetamine may affect how the defendant is initially charged or ultimately sentenced after conviction. The salt form may additionally provide information as to the manufacturing method the operator was using. Again, the chemist must refer to the statutes he is working under to determine whether or not a salt determination is necessary.

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The differentiation of pure methamphetamine base and a methamphetamine salt, usually HCl, is straightforward. The freebase form of methamphetamine is an oily liquid. The HCl salt form is a crystalline solid. The freebase is soluble in most organic solvents. The HCl salt is a solid and is soluble in common solvents like methanol and chloroform but is insoluble in ether, acetone, Freon, and hexane.

The simplest way to determine the salt form of methamphetamine in a volatile organic solvent is to evaporate the solvent and examine the residue. If the residue is a liquid, chances are that the freebase form is present. If a solid residue remains, some sort of salt form is indicated. In either case, IR analysis would confirm the methamphetamine and identify the salt form, if the sample is not contaminated with reaction by-products.

Practical Application 7: Methamphetamine Extraction

Methamphetamine salts are soluble in acidic aqueous solutions, i.e., reaction mixtures. There are times when the salt form of methamphetamine can be used to establish a manufacturing route. The following extraction can be used to remove methamphetamine HI from a reaction mixture without altering the salt form:

Wash an acidic aqueous liquid, or red reaction sludge with ether to remove many of the neutral organic by-products.
Wash the sample with chloroform to remove the mineral acid salts of methamphetamine that may be soluble in chloroform.
Isolate and evaporate the chloroform.
Analyze the residue for methamphetamine and its associated salt form via IR.

Practical Application 8: Ephedrine/Pseudoephedrine Separation

Ephedrine and pseudoephedrine can be reduced to methamphetamine. Their GC retention times and their mass spectra are essentially the same, under the conditions generally used in forensic drug identification. Without derivatization, they are generally considered indistinguishable. Most other screening tests cannot differentiate the two. Their solubility differences can be used to separate them (pseudoephedrine HCl is soluble in chloroform, ephedrine HCl is not). The following extraction can be used to separate pseudoephedrine HCl from ephedrine HCl:

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Dry wash the powdered mixtures of ephedrine HCl and pseudoephedrine HCl with chloroform.
Analyze the dry-washed solid via IR for ephedrine HCl.
Isolate and evaporate the chloroform.
Analyze the residue via IR for pseudoephedrine HCl.

Practical Application 9: Methamphetamine By-Product Profile Extraction

Powdered methamphetamine samples can be a wealth of information, concerning not only the salt form of the methamphetamine but also the presence of reaction by-products and any adulterant or diluents that may be present. The following is a series of extractions that can be used on powdered methamphetamine samples:

Dry wash the sample with acetone to remove niacinamide and reaction by-products.
Isolate the acetone.
Add hexane to the isolated acetone to precipitate out any existing niacinamide.
Isolate and analyze the precipitate by IR.
Analyze the acetone/hexane mixture for the reaction by-products by GCMS or GCFTIR.
Dry wash the solid sample with chloroform to remove the methamphetamine salts.
Isolate and evaporate chloroform.
Dry wash residue with acetone.
Analyze residue by IR to confirm methamphetamine and determine the salt form.
Dry wash the sample with methanol.
Isolate and evaporate the methanol.
Analyze the residue for ephedrine.

Practical Application 10: Quantitation

Determination of the actual amount of controlled substance in a sample is not generally required to establish the facts of a case. However, this knowledge provides investigators and prosecutors with information that can be used to develop investigative leads or to demonstrate what portion of the process the operation was in at the time of seizure. Quantitation information can also

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be used as part of a quality control mechanism within the laboratory. The quantitation method used depends on the information desired and the level of accuracy required. In this section, how different quantitation methods can be employed will be described.

In one application, the concern is determining the amount of pure controlled substance at the time of seizure. As a general rule, the analytical chemist only sees a representative sample of an exhibit. To determine the amount of controlled substance that was in the original container, he must determine the concentration of the sample presented to him. He then uses this value and the original volume to calculate the amount of controlled substance in the original container.

Practical Application 11: Gravimetric Quantitation

To obtain the concentration of the sample, the chemist begins with a 10 ml sample. He performs a series of extractions to isolate the previously identified methamphetamine as the hydrochloride salt. He then divides the weight of the resulting residue (0.10 g) by the volume of the sample to obtain the concentration. The concentration value of the sample can then be multiplied by the original volume of liquid of the item seized (1000 ml) to obtain the amount of methamphetamine in the original container. The following is the resulting calculation sequence:

$$\text{Concentration}_{\text{sample}} = \text{Weight}_{\text{extracted methamphetamine}}/\text{Volume}_{\text{sample}}$$
$$= 0.10\text{ g}/10\text{ ml} = \mathbf{0.010\ g/ml}$$

$$\text{Weight}_{\text{original container}} = \text{Original Volume} * \text{Concentration}_{\text{sample}}$$
$$= 1000\text{ ml} * 0.010\text{ g/ml} = 10.0\text{ g}_{\text{in original container}}$$

Practical Application 12: Serial Dilution Quantitation

Plotting a graph of the sample concentration versus the instrumental response of a gas chromatograph produces a line that can be used to calculate the concentration of an unknown sample. This calculation technique is demonstrated by using the following scenario.

A series of three solutions with a known concentration of Compound *A* are prepared. Their concentrations are 3 mg/ml, 2 mg/ml, and 1 mg/ml, respectively. A 3 mg/ml solution of the questioned sample, which contained an unknown amount of Compound *A*, was also prepared. The peak areas from the gas chromatograph analysis were 3000, 2000, 1000, and 1275, respectively. The concentration of the unknown mixture can be extrapolated

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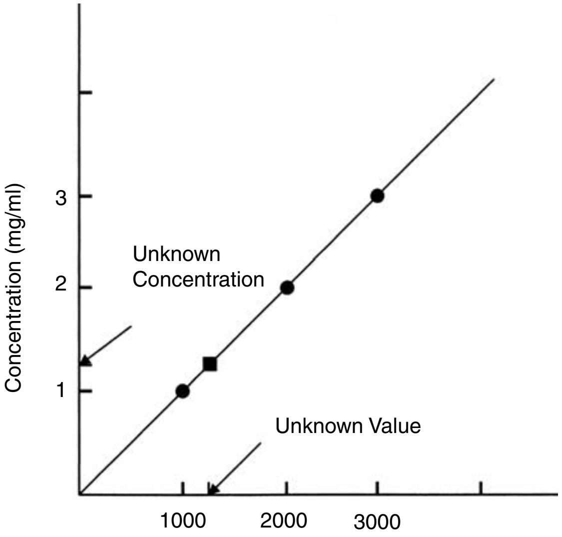

Figure 9.22 Beer's law plot.

directly from the graph. The accuracy and precision of this method is subject to the size of the graph paper and the eye of the chemist (Figure 9.22).

Practical Application 13: Mathematic Application of Serial Dilution Quantitation

The modern gas chromatographs produce precise analytical data that generate a linear response on a graph of concentration versus instrument response. The line tends to travel through the origin, making use of mathematical calculations an option. Therefore, the mathematical equation of a line ($y = mx + b$) can be used to calculate the value of the concentration of the unknown. This removes the subjectivity introduced by the size of the graph paper and the thickness of the pencil that are used to chart the concentration in the previous example.

The first step is to establish the slope of the line (the m value), which is the difference in concentration (the rise) divided by the difference in instrumental response (the run). Using the extreme values of the standard solution, provide a concentration range within which the unknown sample will most likely fall. Using the data from the previous application, the slope calculation becomes the following:

$$\text{Slope} = (\text{Concentration}_{max} - \text{Concentration}_{min})/$$
$$(\text{GC Response}_{max} - \text{GC Response}_{min})$$
$$= (3\ \text{mg/ml} - 1\ \text{mg/ml})/(3000 - 1000) = 0.001\ \text{mg/ml}$$

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The concentration of the unknown can now be calculated by inserting the instrumental data of the unknown solution into the basic equation of the line, as follows:

$$\text{Concentration (y)} = \text{line slope (m)} * \text{peak area (x)} + \text{Y intercept (b)}$$
$$= 0.001 \text{ mg/ml} * 1275 + 0 = 1.275 \text{ mg/ml}$$

The percentage of the mixture that contains the compound is a ratio of the amount of substance in the sample divided by the amount of the original sample. The ratio can be of the weights, as in gravimetric quantitations, or concentrations, as in this instance. In either case, using the data from the above example, the calculation is as follows:

$$\text{Percentage} = (\text{Concentration}_{\text{substance}}/\text{Concentration}_{\text{original sample}}) * 100$$
$$= (1.275 \text{ g/ml}/3.000 \text{ g/ml}) * 100 = 42.5\%$$

Practical Application 14: Single Standard Solution

The precision and linear response of modern instrumentation can be used to further simplify the quantitation process. The quantitation process can be simplified into a single calculation using data from the unknown sample and a single reference standard. By utilizing the premise that the ratio of the sample's concentration and its instrumental response is a constant and is relative to the concentration of the substance, the quantitation calculation can be written as follows:

$$\text{Concentration}_{\text{unknown}} = (\text{Area}_{\text{unknown}} * \text{Concentration}_{\text{standard}})/\text{Area}_{\text{standard}}$$

When the calculation is performed utilizing this relationship and the data from the previous application, the same result is obtained:

$$\text{Concentration}_{\text{unknown}} = 1275 * 3 \text{ mg/ml}/3000 = 1.275 \text{ mg/ml}$$

Using an internal standard in the test solutions increases the accuracy of the results as well as introduces a quality control step into the analytical method. If the same internal standard solution is used to prepare the unknown sample and reference standard, the concentration calculation can be written as follows:

$$\text{Concentration}_{\text{unknown}} = (\text{Area}_{\text{unknown}} * \text{Area}_{\text{internal standard of standard}}$$
$$* \text{Concentration}_{\text{standard}})/(\text{Area}_{\text{standard}} * \text{Area}_{\text{internal standard of unknown}})$$

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The following concentration calculation demonstrates the effect of introducing an internal standard. The only additional information required is the peak area for the internal standards for the reference standard and the unknown solutions, which were 1510 and 1525, respectively.

$$\text{Concentration}_{\text{unknown}} = (1275 * 1510 * 1 \text{ mg/ml})/(1000 * 1525)$$
$$= 1.262 \text{ mg/ml}$$

The 1% difference between the two methods is due to the use of the internal standard. The values obtained in the initial analysis were subject to variations due to sample volumes. The use of an internal standard compensates for variations in injection volumes and will provide a more accurate representation of the actual concentration of the solution.

Practical Example 25: Opinions (Knowledge and Experience)

The opinions provided by a trained clandestine lab chemist can be utilized during any portion of the investigation or prosecution of a clandestine lab operation. As previously stated, a clandestine lab chemist has knowledge beyond that of the traditional forensic chemist. Both the forensic chemist and the clandestine lab chemist must know the limitations of their training and experience and should not render opinions beyond that scope. The following are examples of the effects the opinions provided by chemists have upon various clandestine lab investigations or prosecutions.

A clandestine lab investigator in the southwestern United States came to a senior forensic drug chemist for an opinion concerning the use of lead acetate in the production of phenylacetone. The chemist responded that lead acetate could not be used to produce phenylacetone. The reaction required sodium acetate. This was a true statement in that the synthesis route of choice used sodium acetate in a reaction with phenyl acetic acid. However, he was not a clandestine lab chemist and was not aware of an alternative reaction used in the Pacific Northwest that utilized lead acetate and phenyl acetic acid to produce phenylacetone. The forensic chemist was an excellent bench chemist. However, he did not possess the knowledge and experience necessary to render the required opinion.

Practical Example 26: Opinions (Knowledge and Experience)

Two experts were providing testimony concerning a clandestine lab that was located in a remote desert location. The prosecution's witness held a B.S.

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degree in chemistry and had over 10 years of experience working with clandestine drug laboratories. The defense's expert held a Ph.D. and was a respected former forensic laboratory director who published extensively. However, he had never been directly involved with a clandestine drug lab case, and his publications had done more than mention that certain types of drugs were produced in clandestine drug labs. During the verdict portion of the bench trial the judge commended the defense expert's service to forensic sciences. He then pronounced his testimony unbelievable. His opinion was valid. However, it was based on his reputation and not on his actual experience and training in the area of clandestine labs.

Practical Application 15: Opinions (Data Interpretation)

Volumes of technical information can be generated during a clandestine lab investigation. It is the job of the clandestine lab chemist to extract the relevant information and place it into some semblance of order. The following are examples of how a clandestine lab chemist brought calm to the chaos by using the physical evidence to answer some of the "who, what, when, where, why, and how" questions that required answers.

The following chemicals were found at the scene of a suspected clandestine drug lab: acetic acid, acetic anhydride, aluminum powder, bromobenzene, hydrochloric acid, mercuric chloride, methylamine, nitroethane, phenylacetic acid, and sodium acetate. The expert was asked to determine what the most likely final product of the operation was based on the chemicals found at the scene. A sequential evaluation of the potential manufacturing routes allowed the expert to establish the manufacturing route the operator most likely was implementing.

The evaluation of the chemicals begins by making a table with two columns. In one column, list the chemicals found at the scene. In the other column, list the potential products that could be manufactured and the chemical's role in the process (i.e., solvent, reagent, or precursor). The chemist should then have a table that correlates chemicals to their potential end products. Appendix C of this book was provided to be used for this purpose. The Chemistry Guide of the DEA Clandestine Laboratory Training manual is also a source of this information.

A pattern becomes apparent once all of the chemicals have been assigned a potential end product. At this point, the expert looks at individual synthesis routes to establish if all, or a significant portion, of the chemicals are present. Chemicals for multiple synthesis routes may be present. Many times, there may be chemicals present that have nothing to do with the synthesis being

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Table 9.1

Chemical	Product/Route
Acetic acid	rA1 rMD1, 2, 5 pP4
Acetic anhydride	pP1
Aluminum	rA1 rM1
Bromobenzene	pPC1, 2, 3
Hydrochloric acid	rAll A, M, MD, PC
Mercuric chloride	rM1
Methylamine	pMD3, 4 pM1,4,5
Nitroethane	pA2 pMD2 pP5
Phenylacetic acid	pP1, 3, 4
Sodium acetate	rP1

Note: A = amphetamine, M = methamphetamine, MD = MDA, P = phenylacetone, PC = phencyclidine, # = synthesis route number, p = precursor, r = reagent.

used. The likely final product is the one with the most complete set of the required chemicals.

The information compiled in Table 9.1 indicates that the most probable end products from the list of chemicals are phenylacetone or methamphetamine. In one step, the expert narrowed the field of possible final products from all controlled substances that are commonly produced in clandestine labs to two.

Once the field of possible final products is narrowed to a manageable number, the expert compares the list of known chemicals to the list of precursor and reagent chemicals required for each of the various synthesis routes (Appendix C). In this example, the list of chemicals supports two different synthesis routes. One route suggests the production of phenylacetone using a phenylacetic acid, sodium acetate, and acetic anhydride. The other suggested synthesis route produces methamphetamine using phenylacetone, methylamine, aluminum, and mercuric chloride. Each method supports the existence of the other. At the time of this seizure, the manufacturing method of choice for the production of methamphetamine was a two-step process that used phenylacetone as an intermediate product.

Practical Application 16: Opinions (Data Interpretation)

The complete analysis of a reaction mixture from an operational clandestine lab produces a volume of information concerning the method the operator was utilizing to manufacture the controlled substance involved. The thorough analysis of the data from any given analytical technique may be all that is required to profile the synthesis route being used. The following is an example

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Table 9.2 Reaction Mixture Components

Compound	Peak 1	Peak 2	Peak 3	Peak 4	Peak 5	Mole. Wt.	Drug/Synthesis Route*
Ephedrine	58	69	79	41	59	165	M2, M3
Phenyl-2-propanone	91	134	92	43	65	134	Numerous A and M routes
1,2-Dimethyl-3-phenylaziridine	146	105	42	132	91	147	M2, M3
1-Benzyl-3-methylnaphthalene	232	217	108	215	202	232	M3, A1
1,3-Dimethyl-2-phenylnaphthalene	232	215	217	108	202	232	M3, A1

* See Appendix D.

of how the mass spectral analysis of a reaction mixture sample can be used to determine the synthesis route.

The mass spectral analysis of a clandestine lab reaction mixture produced six significant peaks in addition to the detected controlled substance, methamphetamine (Table 9.2). Each of the compounds was tentatively identified by a database search of the five most prominent ions in their mass spectrum (Appendix K). The database also indicated which manufacturing methods were associated with each compound. The pattern that emerged from the evaluation of the potential manufacturing routes indicated that the operator was converting ephedrine to methamphetamine using the hydriodic acid reduction technique.

Practical Application 17: Opinions (Production Estimates)

How much controlled substance could the operation produce is a question that will always be asked at some point during the investigation or prosecution. The production amount may or may not be an element of the crime, but it may be significant during the prosecution or the sentencing phase if a conviction is obtained.

The expert should routinely calculate the operation's estimated production as one of his opinions. The information to determine these production estimates is readily available if the lab scene was documented properly. The value that is relevant may be debatable. Is the amount of controlled substance that could be produced with the chemicals on hand the benchmark figure? Or, should the amount of finished product that could be produced if the operator had all the chemicals necessary to completely use the chemicals found at the site the appropriate value? This philosophical difference in opinion necessitates that the expert calculate a range. The following are

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examples of calculations used to determine the production of various controlled substances.

The expert needs to know the amount of phenylacetone that can be produced from 1000 g of phenylacetic acid. He first calculates the reaction's conversion factor (*n*) by dividing the molecular weight of the phenylacetone (the product) by the molecular weight of the phenylacetic acid (the reactant). In Appendix N, the conversion factors for chemicals and drugs most commonly encountered in clandestine labs are presented.

$$n = \text{Molecular weight phenylacetone/Molecular weight phenylacetic acid} = 134/136 = 0.98$$

The weight of the phenylacetic acid (precursor chemical) is multiplied by the conversion factor to produce the weight of the phenylacetone (final product) at 100% conversion:

$$\text{Weight phenylacetone}_{\text{theoretical}} = n * \text{Weight phenylacetic acid} = 0.98 * 1000\text{ g} = 980\text{ g}$$

In some instances, the precursor is in a solution. The lower concentration must be accounted for in the production calculation. For example, methylamine is commonly found on a 40% (weight/volume) aqueous solution. The following example illustrates the modifications necessary to account for a diluted solution of 1000 g of a methylamine solution used as a precursor chemical.

$$\text{Weight methamphetamine HCl}_{\text{theoretical}} = n * \text{Volume}_{\text{methylamine}} * \text{Dilution factor} = 5.96 * 1000\text{ ml} * 0.40\text{ g/ml} = 2384\text{ g}$$

Practical Application 18: Opinions (Production Estimates, Multistep)

Multistep reactions contain an intermediate that must be accounted for. Each step of the reaction has a conversion factor that figures into the final calculation. In these instances, the calculation boils down to a sequence of single-step calculations that use the weight of the previous calculation as the starting point for the next in the sequence. The intermediate acts as the product in one calculation, and the precursor does in the next. The calculated weight of the intermediate is used as the precursor weight for the second step of the process.

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The benzyl cyanide synthesis of phenylacetone and subsequent conversion to methamphetamine HCl can be used to demonstrate the calculation sequence. The conversion factors for both steps can be calculated as in the previous examples or can be taken from a table of precalculated values:

$$n_1 = \text{Molecular weight phenylacetone/Molecular weight benzyl cyanide} = 134/117 = 1.14$$

$$n_2 = \text{Molecular weight methamphetamine HCl/Molecular weight phenylacetone} = 185/134 = 1.38$$

The weight of the phenylacetone intermediate is calculated as an independent step. Using the standard 1000 g of benzyl cyanide as a starting point, the calculation is as follows:

$$\text{Weight phenylacetone}_{\text{theoretical}} = n * \text{Weight benzyl cyanide} = 1.14 * 1000\text{ g} = 1140\text{ g}$$

$$\text{Weight methamphetamine HCl}_{\text{theoretical}} = n_2 * \text{Weight phenylacetone}_{\text{theoretical}} = 1.38 * 1140\text{ g} = 1573\text{ g}$$

Practical Application 19: Opinions (Per Batch Production Estimates)

The per batch estimate can be a significant point of debate. The potential of an operation to produce 10 kg of controlled substance loses its significance if it can only be produced in 10 g batches due to limitations placed upon it by the size of the available equipment. The following is an example of estimating the per batch production of an operation, using the equipment as a limiting factor.

The expert is asked to calculate the amount of methamphetamine that could be produced using a 1000 ml reaction flask. Without additional information, the expert makes the following assumptions. First, he uses a common chemical ratio for the methamphetamine reaction, which is 4 l of acid, 1 kg precursor, and 500 g of an additional reagent. He also assumes a reaction mixture volume of 2/3 the total volume of the reaction flask. His calculations are as follows:

$$\text{Volume}_{\text{reaction mixture}} = \text{Volume}_{\text{flask}} * 66\% = 1000\text{ ml} * 0.66 = 660\text{ ml}$$

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$$\text{Reaction ratio} = \text{Precursor amount/Acid amount}$$
$$= 1000 \text{ g}/4000 \text{ ml} = 0.25 \text{ g/ml}$$

$$\text{Weight precursor}_{\text{reaction mixture}} = \text{Reaction ratio} * \text{Volume}_{\text{reaction mixture}}$$
$$= 0.25 \text{ g/ml} * 660 \text{ ml} = 165 \text{ g}$$

$$\text{Weight product}_{\text{theoretical}} = n * \text{Weight precursor}_{\text{reaction mixture}}$$
$$= 0.92 * 165 \text{ g} = 152 \text{ g}$$

Practical Example 27: Testimony

The following is an example of the need for understanding the definitions of the terms that are used during the testimony. The original exchange was between a defense attorney and the State's expert during a controlled substance trial. This exchange could easily have happened between an attorney and his own expert without pretrial preparation.

Attorney: Mr. Chemist, did you perform a qualitative analysis on Exhibit A?
Chemist: Yes sir.
Attorney: What percentage of Exhibit A contained a controlled substance?
Chemist: I did not perform that examination.
Attorney: Did you perform a qualitative analysis on Exhibit A?
Chemist: Yes sir.
Attorney: And what percentage of Exhibit A contained a controlled substance?
Chemist: I did not perform that examination.
Attorney: Mr. Chemist, you said you performed a qualitative analysis on Exhibit A?
Chemist: Yes sir.
Attorney: Then what percentage of Exhibit A contained a controlled substance?
Chemist: I did not perform that examination.
Attorney: Mr. Chemist, why won't you answer my question?
Chemist: Sir, I am trying to use proper terminology, as you requested.

This exchange demonstrates that the attorney clearly did not understand the difference between qualitative (what is it?) analysis and quantitative (how much is there?) analysis. The chemist's responses clearly created an adversarial atmosphere by specifically answering the question posed. He could have

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provided the information the attorney desired in a less combative style, placing him in a better light with the jury.

Practical Example 28: Testimony

The chemist analyzed a 10 bail representative sample of a 200 bail seizure of marijuana. The pretrial preparation consisted of brief introductions. Below is an excerpt of the resulting testimony.

Attorney: Mr. Chemist, I show you Exhibit A. Do you recognize it?
Chemist: No sir, I do not. (The chemist was presented a 50-lb, plastic-wrapped bail of plant material. He looked and did not find the identifying marks he placed on it at the time of analysis. He expected to be presented one of the 10 bails he actually analyzed.)
Attorney: **Silence.** (The attorney expected a Yes, which would lead into his next question.)
Attorney: Do you recognize anything on Exhibit A?
Chemist: Yes sir, I recognize the case number.
Attorney: Where do you recognize that case number?
Chemist: From a submission I analyzed in July of this year.
Attorney: What did that submission consist of?
Chemist: Ten plastic wrapped bails of plant material.
Attorney: Did those exhibits resemble Exhibit A?
Chemist: Yes sir.
Attorney: Did you analyze the exhibits submitted to you in July of this year?

The testimony continued. The results of the analyses of the representative samples were eventually allowed into the record.

This whole exchange could have been avoided and boiled into a few flowing questions. Do you recognize the exhibit and how? Did you analyze the exhibit? What are the results of your analysis? However, the lack of proper preparation resulted in both parties being surprised, resulting in additional questions and answers to establish the same facts.

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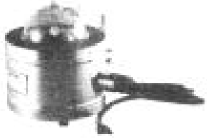
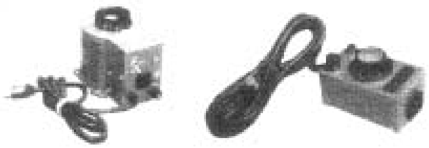
Appendix A

Scientific Equipment Encountered at Clandestine Labs

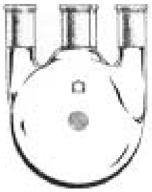

Hard Side

Soft Side

Heating Mantles

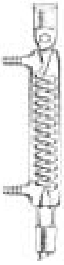

Heating Mantle Controls

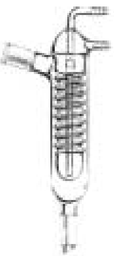
Single Neck


Triple Neck

Reaction Flasks

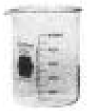
Allihn


Graham


Friedich

Liebig

Condensers


Beaker

Reflux

Simple Distillation

Fractional Distillation

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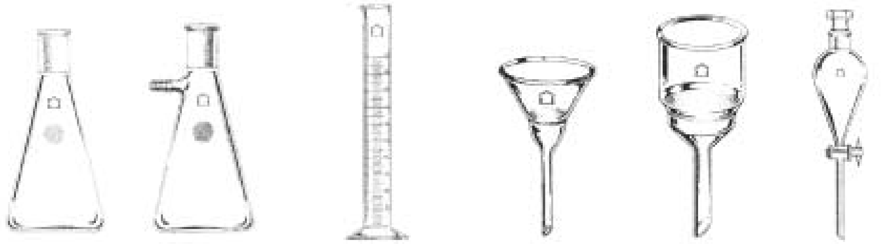
Scientific Equipment Encountered at Clandestine Labs (Continued)



Erlenmyer | Vacuum | Graduated Cylinder | Filter | Buchner | Separatory

Flasks | **Funnels**

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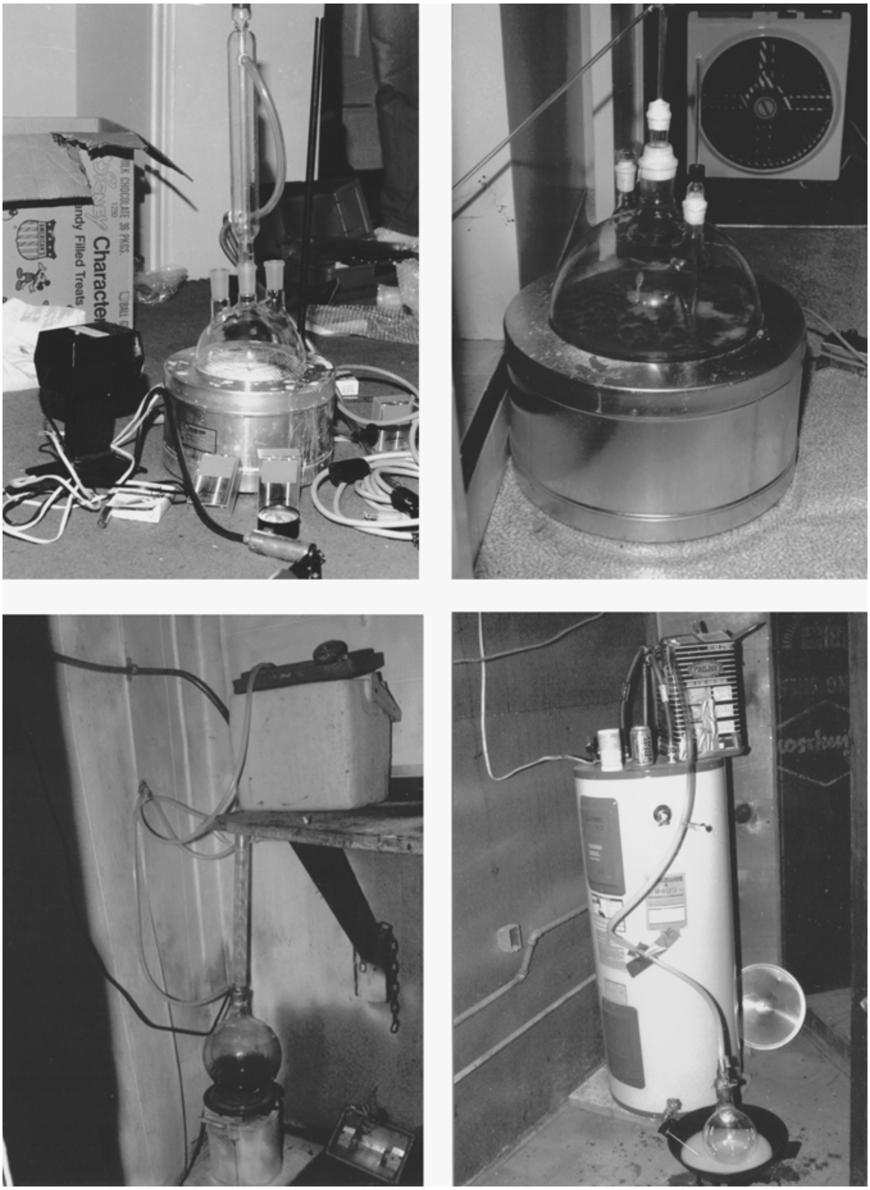
Reflux Variations



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Distillation Variations

Hydrogenator Variations

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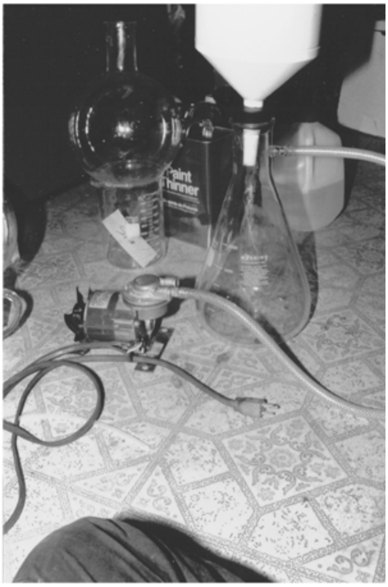
Vacuum Filtration Variations



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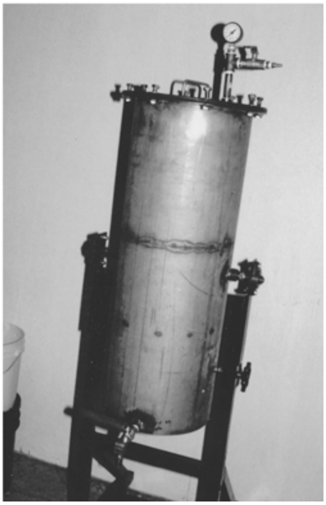
Extraction Equipment Variations



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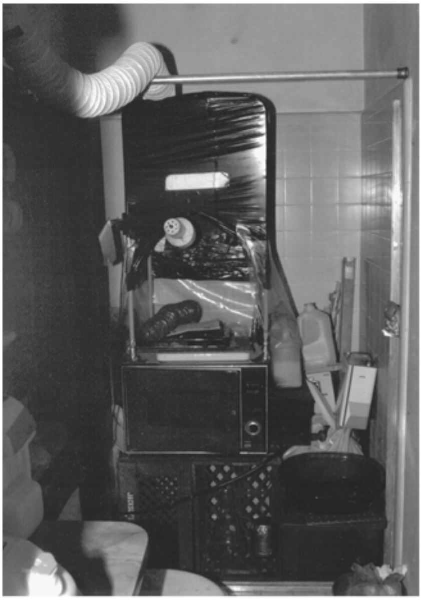
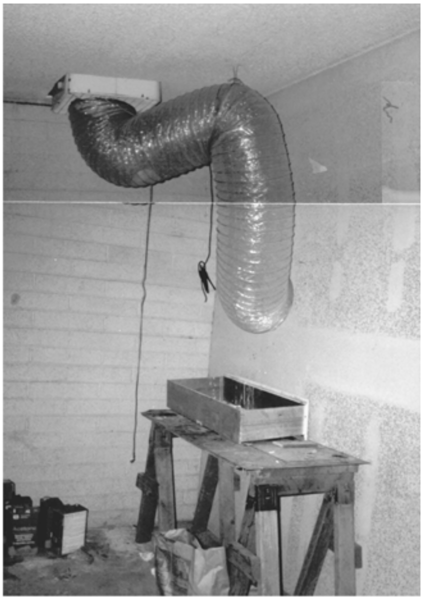
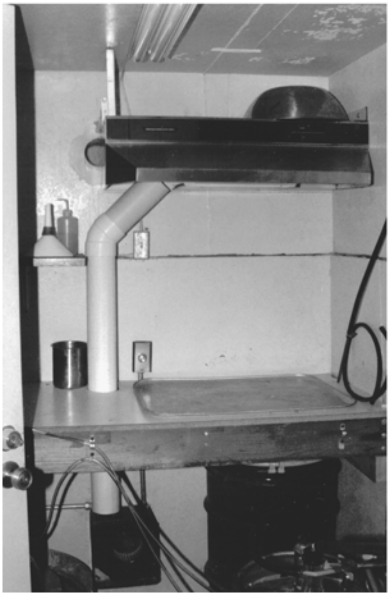
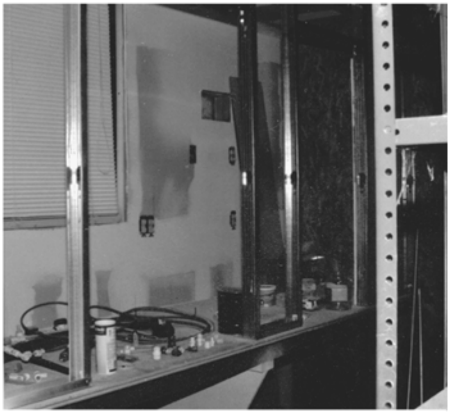
Makeshift Ventilation

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Appendix B: Legitimate Use Table

Chemical	Legitimate Uses
Acetaldehyde*	Perfumes, flavors, plastics, silver mirrors
Acetic acid*	Food preservative, organic synthesis
Acetic anhydride*	Organic synthesis, dehydrating agent
Acetone	General solvent
Acetonitrile*	Solvent, organic synthesis
Allylbenzene*	None listed
Allylchloride*	Organic synthesis
4 allyl 1,2 methylenedioxybenzene*	None listed
Aluminum powder	Paint additive, pyrotechnics, explosives, metal alloys
Aluminum chloride*	Disinfectant, deodorant, wood preservative
Ammonia gas*	Refrigerant, manufacture of nitric acid and explosives
Ammonium chloride*	Batteries, electroplating, textiles
Ammonium formate*	Inorganic metal analysis
Ammonium hydroxide*	None listed
Ammonium nitrate	Fertilizer, explosives, matches, pyrotechnics
Aniline*	Dyes, perfumes, varnishes, organic synthesis
Benzaldehyde*	Dyes, perfumes, flavors
Barium chlorate	Pyrotechnics, explosives, matches, dye processes
Barium nitrate	Pyrotechnics, explosives, vacuum tube manufacture
Benzene*	Pharmaceuticals, dyes, industrial solvent
Benzyl chloride*	Dyes, pharmaceuticals, perfumes, resins
Benzyl cyanide*	None listed
Bromobenzene*	Grignard reagent
Bromoethane*	None listed
Carbon dioxide	Beverage carbonation, fire extinguishers, dry ice
Carbontetrachloride*	Dry cleaning, fire extinguishers, general solvent
Chloro-2-propanone*	Intermediate for perfumes, drugs, insecticides, photography
Copper oxide	Ceramic and glass pigment, glass polishing
Copper sulfate	Fungicide, photography, food additive, paint pigment

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Chemical	Legitimate Uses
Cyclohexanone*	Industrial solvent
Dichloroethane*	None listed
Ephedrine*	Bronchodilators
Ergotamine tartrate*	Treatment of migraine headaches
Ethyl acetate*	Photo film, perfume, gun powder, dry cleaning
Formamide*	Organic synthesis
Formic acid*	Tanning, electroplating, wool dying
Hydrazine	Reducing agent, rocket fuel
Hydrobromic acid*	Analytical reagent
Hydrochloric acid	Pool chemical, masonry and metal cleaner, mining
Hydrogen	Welding
Hydrogen peroxide*	Rocket fuel, bleaching agent
Hydriodic acid*	Pharmaceuticals, disinfectants
Hydroxylamine HCl*	Photography, antioxidant for soaps
Iodine*	Germicides and antiseptics, catalyst
Lithium metal*	Catalyst, metal alloys, batteries
Lithium aluminum hydride*	Reducing agent
Lithium hydroxide	Photographic developers
Magnesium metal	Metal alloys, pyrotechnics, Grignard reagent
Mercuric chloride	Preservative, photography, mining, steel etching
Mercury*	Thermometers, switches, lighting, mining, dentistry
Methanol	Solvent, antifreeze, gas additive, camping fuel
Methyl acrylate*	Plastics and textiles
Methylamine*	Tanning, organic synthesis
Nitric acid	Manufacture of fertilizers, dyes, explosives, and a variety of organic chemicals
Nitroethane	Fuel additive, solvent
Perchloric acid*	Metal plating, explosives
Phenylacetic acid*	Perfumes
Phenylacetonitrile*	Organic synthesis
Phenylmagnesium bromide*	Organic synthesis
Phosphoric acid*	Fertilizers, detergents, food additive, cleaning solvent
Phosphorous red*	Pyrotechnics, matches, fertilizers, pesticides
Phosphorous pentachloride*	Catalyst
Piperidine*	Organic synthesis
Potassium carbonate	Soap, glass, pottery, engraving, tanning
Potassium chlorate	Explosives, pyrotechnics, matches, dye industry
Potassium chromate	Leather tanning, rust-proofing metal
Potassium cyanide	Mining, electroplating, steel hardening
Potassium dichromate	Leather tanning, dyes, paints, pyrotechnics, matches, waterproofing fabrics
Potassium hydroxide	Paint and varnish remover, photo engraving, printing inks
Potassium iodide*	Oxidizer for chemical analysis
Potassium nitrate	Tempering steel, tobacco curing, glass manufacture, explosives
Potassium perchlorate	Explosives, pyrotechnics, photography
Pyridine*	Solvent, organic synthesis

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Chemical	Legitimate Uses
Raney nickel*	Catalyst
Sodium acetate	Photography, food additive
Sodium bicarbonate	Baking soda, fire extinguishers, cleaning compounds
Sodium bisulfite	Disinfectant, textile bleaching, food additive
Sodium hydroxide	Drain cleaner
Sodium metal*	Sodium compounds, sodium vapor lamps
Sodium nitrate*	Glass manufacturing, pottery, fertilizer, meat and tobacco preparation
Strontium nitrate	Pyrotechnics, road flares, matches
Sulfuric acid	Battery acid, drain cleaner, fertilizers, explosives
Tartaric acid	Food additive, photography, tanning, ceramics
Thorium nitrate*	Catalyst
Trifluoroacetic anhydride*	None listed
Zinc metal	Metal alloys, batteries, mining, printing plates, household utensils

* No legitimate home or hobby use.

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Appendix C: Drug Precursor/ Reagent Table A

Compound	Amphetamine	MDA/ MDMA	Methamphetamine	PCP Analog	P-2-P
Acetic acid	r A2,3	r MD1,2,5			p P4
Acetic anhydride					p P1
Acetaldehyde			p M4		
Acetonitrile	p A6				
α-Acetylphenylaceto-nitrile					p P2
Allyl benzene	p A6				
Allyl chloride	p A5		p M6		
4-Allyl-1,2-methylene-dioxy benzene		p MD1			
Aluminum foil	r A4		r M1		
Aluminum chloride		r MD1,2			r P5,7
Ammonia gas	p A4	r MD3 p MD5	r M(7)		
Ammonium acetate	p A1	r MD2			
Ammonium chloride			r M(7)		
Ammonium formate	p A1	p MD1			
Ammonium hydroxide	p A1			All	
Benzaldehyde	p A2				p P5
Benzene	p A5				p P7
Benzyl chloride			p M4		
Benzyl cyanide					p P2
Bromobenzene				p PC1a,2a,3a	
Bromothiophene				p PC1b,2b,3b	
Butylamine	r A2				
Chloroacetone					p P7
3-Chloropropene	p A5		p M6		
Copper sulfate			r M1		
Cuprus oxide		r MD5			
Cyclohexanone				All	

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Compound	Amphetamine	MDA/ MDMA	Methamphetamine	PCP Analog	P-2-P
Dibromomethane		r MD2			
Ephedrine/ pseudoephedrine			p M2,3,7		
Ethyl acetate					p P2
Formamide	p A1	p MD1			
Formic acid	r A1	r MD4	r M5		
HBr		r MD5			
HCl	r All	r All	r All	r All	
Hydrogen	r A2,3		r M2		
Hydrogen peroxide		r MD1			
HI	r A7		r M3		
Hydroxylamine HCl	p A3				
Iodine			r M3		
Iron fillings					r P5
Isosafrole		p MD1			
Lithium			r M(7)		
Lithium aluminum hydride	r A2,3	r MD2			
Magnesium turnings			r M4	r All	
Manganous carbonate					r P4
Manganous chloride					r P4
Mercuric chloride	r A4	r MD3,4,5	r M1		
Mercury	r A2				
Methylamine		p MD3,4	p M1,4,5		
Methylformamide		p MD4	p M5		
Nitroethane		p MD2			p P5
Norpseudoephedrine	p A7				
Palladium sulfate			r M2		
Perchloric acid			r M2		
Phenylacetic acid					p P1,3,4
Phenylacetonitrile					p P2
Phenylmagnesium bromide				p PC1a,2a,3a	
Phenylacetone	p A1,3,4		p M1,5		
Phosphorous	r A7		r M3		
Phosphorous pentachloride			r M2		
Piperonal		p MD2			
Piperidine				p PC1	
Platinum			r M2		
Platinum chloride			r M2		
Platinum oxide			r M2		
Potassium carbonate				r All	
Potassium cyanide				r All	
Potassium hydroxide	r All	r All	r All	r All	r All
Pumic					r P4
Pyridine				p PC2	
Raney nickel	r A2,3		r M2		
Sodium			r M1		
Sodium acetate					r P1

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Compound	Amphetamine	MDA/ MDMA	Methamphetamine	PCP Analog	P-2-P
Sodium amalgam	r A2,3				
Sodium bisulfite				r All	r All
Sodium hydroxide	r All	r All	r All	r All	r All
Sodium sulfate	r All				r P1,2,5
Sulfuric acid	r All	r MD1	r All		r P2
Thionyl chloride			r M2		

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Appendix C: Drug Precursor/ Reagent Table B

Reaction	Precursors	Reagents
A1	Ammonia Ammonium formate Formamide Phenylacetone	Formic acid Hydrochloric acid Sulfuric acid
A2	Benzaldehyde Nitroethane	Acetic acid Butylamine Hydrogen Lithium aluminum hydride Sodium amalgam Raney nickel
A3	Hydroxylamine HCl Phenylacetone	Acetic acid Hydrogen Lithium aluminum hydride Raney nickel Palladium black Sodium acetate Sodium amalgam
A4	Ammonia Phenylacetone	Aluminum Mercuric chloride
A5	Benzene Allyl chloride Ammonia	Ferric chloride

Continued.

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Reaction	Precursors	Reagents
A6	Allylbenzene Acetonitrile	Hydrochloric acid
A7	Phenylpropanolamine	Hydriodic acid Red phosphorous
MD1	Ammonia Ammonium formate Formamide Isosafrole	Acetic acid Formic acid Hydrochloric acid Hydrogen peroxide Sulfuric acid
MD2	Nitroethane Piperonal	Acetic acid Ammonium acetate Lithium aluminum hydride
MD3	Isosafrole Methylamine	Aluminum foil Mercuric chloride
MD4	Isosafrole Methylamine Methylformamide	Acetic acid Formic acid Hydrochloric acid Hydrogen peroxide Sulfuric acid
MD5	Ammonia (MDA) Methylamine (MDMA) Safrole	Cuprus oxide Hydrobromic acid Mercuric chloride Sodium carbonate Sodium hydroxide
M1	Phenylacetone Methylamine	Aluminum Mercuric chloride
M2	Ephedrine	Hydrogen Palladium black Palladium sulfate Perchloric acid Phosphorous pentachloride Platinum Platinum chloride Sodium acetate Sulfuric acid Thionyl chloride
M3	Ephedrine	Hydriodic acid Iodine Red phosphorous
M4	Acetaldehyde Benzylchloride Methylamine	Iodine Magnesium

Continued.

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Reaction	Precursors	Reagents
M5	Phenylacetone Methylamine Methylformamide	Formic acid Hydrochloric acid
M6	Allylchloride Benzene Methylamine	Ferric chloride
M7	Ephedrine	Ammonia Ammonium chloride Lithium or sodium Sodium chloride Tetrahydrofuran
PC analogs	Bromobenzene Bromothiophene Cyclohexanone Morpholine Phenylmagnesiumbromide Piperidine Pyridine Pyrolodine	Magnesium Potassium cyanide Sodium cyanide
P1	Phenylacetic acid Acetic anhydride	Sodium acetate
P2	Benzylcyanide Ethyl acetate	Acetic acid Phosphoric acid Sodium Sulfuric acid
P3	Phenylacetic acid	Lead acetate
P4	Phenyl acetic acid Acetic acid	Hydrochloric acid Manganous carbonate Manganous chloride Nitric acid Pumic Sodium carbonate Thorium nitrate
P5	Benzaldehyde Nitroethane	Butylamine Ferric chloride Iron filings
P6	Phenyl-2-propanol	Potassium dichromate
P7	Benzene Chloroacetone	Aluminum chloride Sodium bisulfate

Continued.

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Drug Precursor/Reagent Table Methods Key

A1	Amphetamine via Leuckart reaction
A2	Amphetamine via benzaldehyde/nitroethane
A3	Amphetamine via P-2-P/hydroxylamine
A4	Amphetamine via P-2-P/ammonia
A5	Amphetamine via benzene/allyl chloride/ammonia
A6	Amphetamine via allylbenzene/acetonitrile
A7	Amphetamine via phenylpropanolamine/HI
MD1	MDA via isosafrole to 3,4-methylenedioxy P-2-P, using the Leuckart reaction
MD2	MDA via piperonal/nitroethane
MD3	MDMA via 3,4-methylenedioxy P-2-P/methylamine
MD4	MDMA via 3,4-methylenedioxy P-2-P using the Leuckart reaction
MD5a	MDA via safrole/HBr/ammonia
MD5b	MDMA via safrole/HBr/methylamine
M1	Methamphetamine via P-2-P/methylamine
M2	Methamphetamine via ephedrine/H_2
M3	Methamphetamine via ephedrine/HI
M4	Methamphetamine via benzyl chloride/acetaldehyde/methylamine
M5	Methamphetamine via Leuckart reaction
M6	Methamphetamine via benzene/allyl chloride/methylamine
M7	Methamphetamine via Birch reduction
PC1	Piperidine/cyclohexane intermediate
PC1a	Phenyl addition
PC1b	Thiophene addition
PC2	Pyridine/cyclohexane intermediate
PC2a	Phenyl addition
PC2b	Thiophene addition
PC3	Morpholine/cyclohexane intermediate
PC3a	Phenyl addition
PC3b	Thiophene addition
P1	P-2-P via phenylacetic acid/acetic anhydride
P2	P-2-P via benzyl cyanide/ethyl acetate
P3	P-2-P via phenylacetic acid/lead acetate
P4	P-2-P via phenylacetic acid/acetone
P5	P-2-P via benzaldehyde/nitroethane
P6	P-2-P via phenyl-2-propanol/dichromate
P7	P-2-P via benzene/chloroacetone

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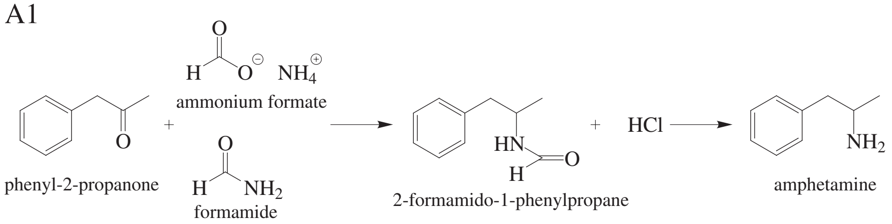
Appendix D: Reaction Mechanisms

Amphetamine Reactions

A1



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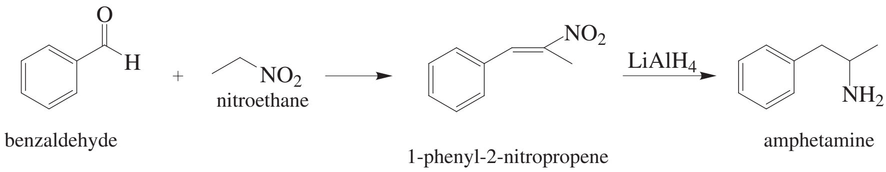

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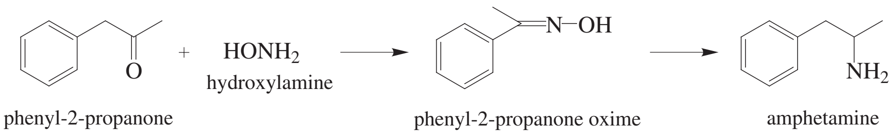

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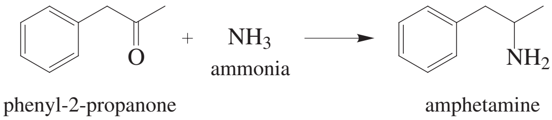
A4



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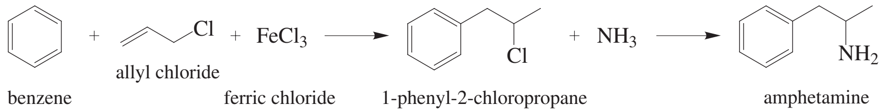

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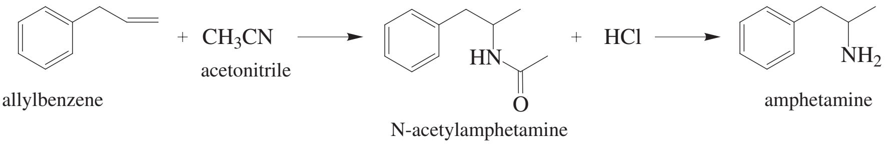

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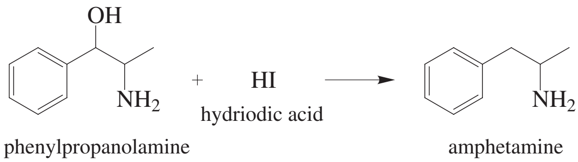
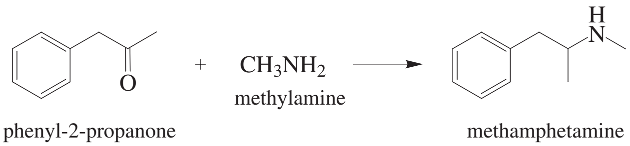


Methamphetamine Reactions

M1



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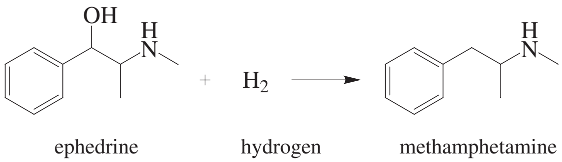
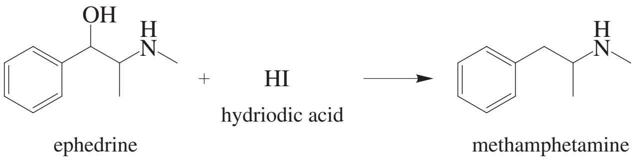
M2



M3



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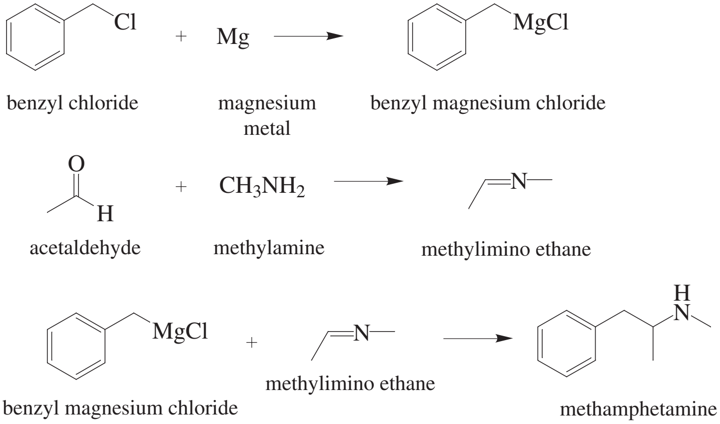
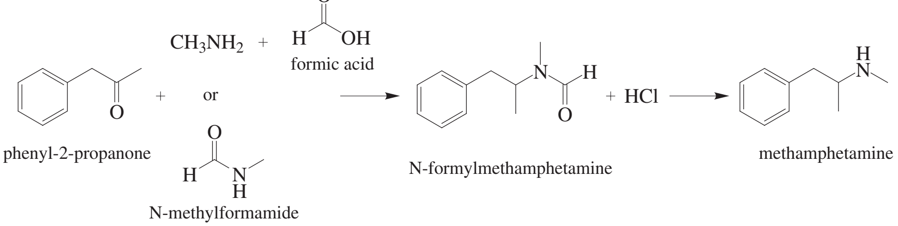


M5



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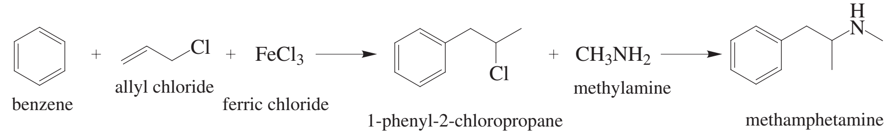
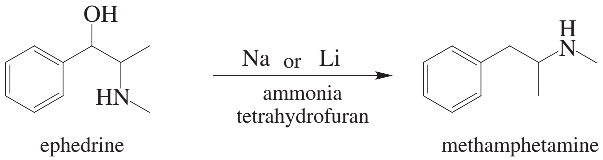
M6

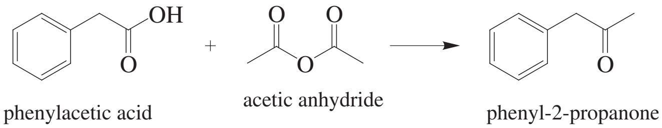


M7



Phenylacetone Reactions

P1



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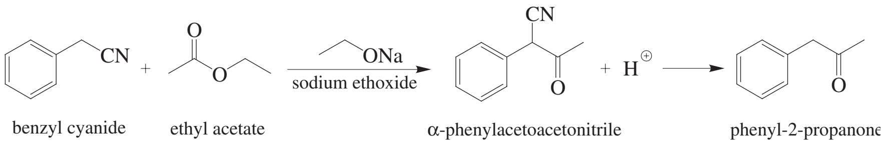
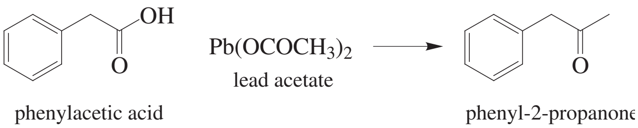


P3



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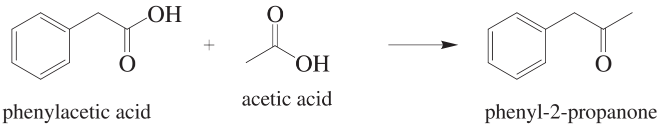
P4



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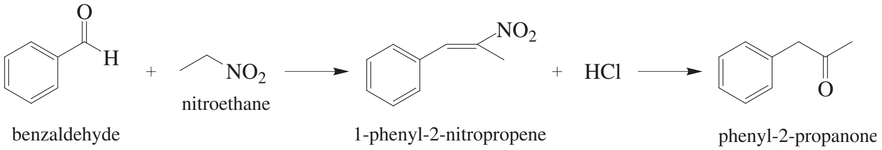

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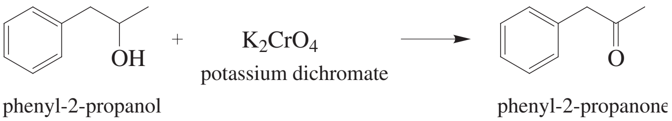

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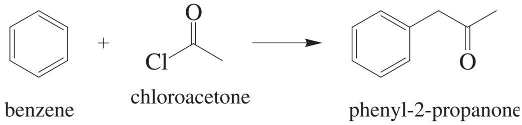
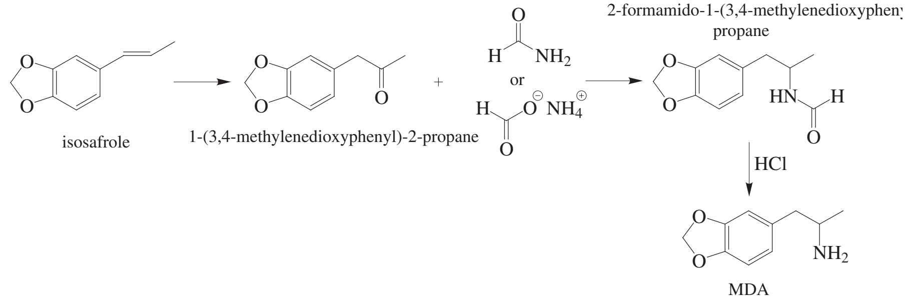


MDA/MDMA Reactions

MD1



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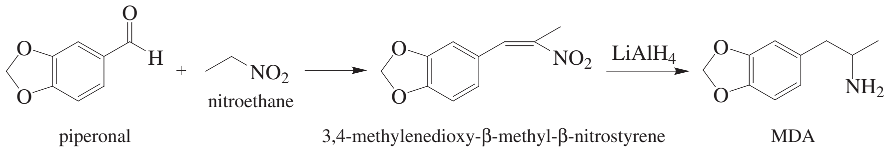
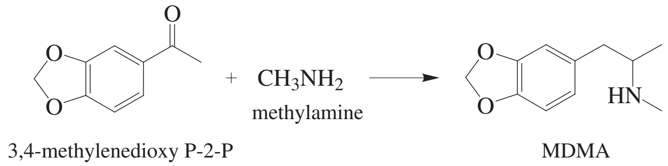
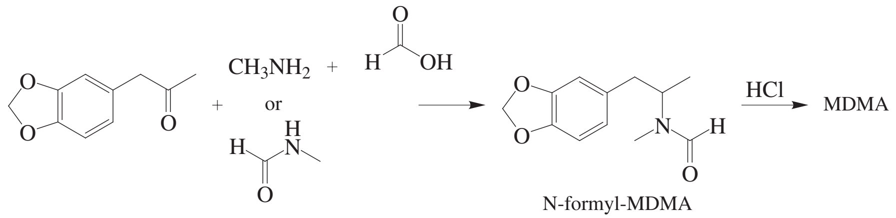
MD2



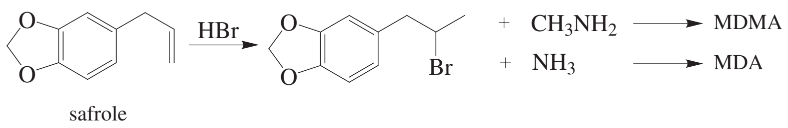
MD3



MD4

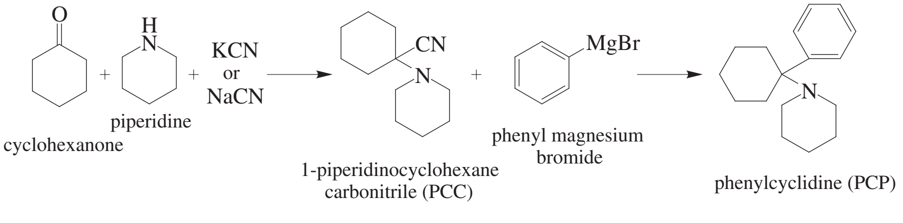


MD5



Phencyclidine Analogs

PC1



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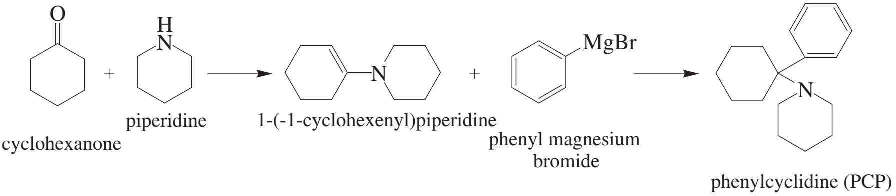
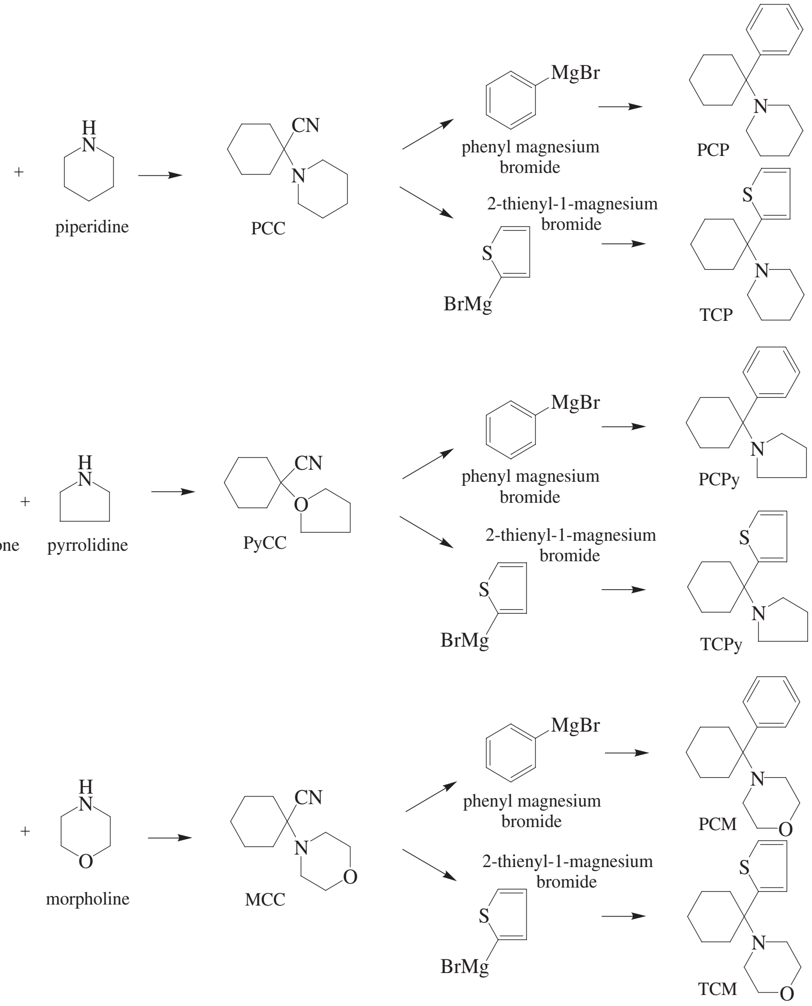
PC2





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Appendix E: Chemical Hazards

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Chemical	Hazards (NFPA Rating: H/F/R)	Flash Point (°F)	Explosive Limit (Lower/Upper)	Incompatibilities
Acetaldehyde	3/4/2	–6	4%/60%	Strong oxidizers, acids, bases, alcohols, ammonia and amines, phenols, ketones, HCN, H_2S [Note: Prolonged contact with air may cause formation of peroxides that may explode and burst containers; easily undergoes polymerization]
Acetic acid	3/2/0	104	4%/19.9%	Metals; acetic anhydride; alcohols; amines; ammonium nitrate; chlorine trifluoride; nitric acid; permanganates; peroxides; sodium hydroxide; sodium peroxide; hydrogen peroxides; acetaldehyde; caustics (e.g., ammonia, ammonium hydroxide, calcium hydroxide, potassium hydroxide, sodium hydroxide); acid anhydrides; chlorosulfonic acid; oleum; chromium trioxide; potassium hydroxide; carbonates; bromine pentafluoride; perchloric acid; chromic anhydride; potassium-*tert*-butoxide; calcium salts; ethyleneimine; attacks some forms of plastics, rubbers, and coatings; 2-aminoethanol; ethylene diamine; phosphorus trichloride; chromic acid anhydride; phosphorus isocyanate; diallyl methyl carbinol + ozone; nitric acid + acetone; xylene; sodium salts
Acetic anhydride	3/2/1	NA	2.9%/10.3%	Strong oxidizing agents, strong reducing agents, bases, alcohols, metal powders, moisture
Acetone	1/3/0	–4	2.5%/12.8%	Strong oxidizing agents, strong acids, perchlorates, aliphatic amines, chromyl chloride, hexachloromelamine, chromic anhydride, chloroform + alkali, potassium *tert*-butoxide
Acetonitrile	2/3/0	–4	4.4%/16%	Oxidizing agents; reducing agents; acids; bases; alkali metals; fluorine; nitric acid; perchlorates; sulfuric acid; chlorosulfonic acid; oleum; dinitrogen tetraoxide; sulfites; indium; moisture; attacks some forms of plastics, rubbers, and coatings; nitrating agents; N-fluoro compounds (e.g., perfluorourea + acetonitrile); lanthanide perchlorates; iron (III) perchlorate; 2-cyano-2-propyl nitrate; trichlorosilane; diphenyl sulfoxide
Allylbenzene	NA	177	NA/NA	NA

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Allylchloride	2/3/1	30	2.9%/11.1%	Explosion hazard when exposed to acids or oxidizing agents; explosive reaction with alkyl aluminum chlorides + aromatic hydrocarbons (e.g., benzene or toluene); violently exothermic polymerization reaction with Lewis acids (e.g., aluminum chloride, boron trifluoride, and sulfuric acid); incompatible with ethylene imine, ethylenediamine, chlorosulfonic acid, oleum, sodium hydroxide, and nitric acid
Aluminum chloride	3/0/2	NA	NA/NA	Water; organic materials; aluminum chloride reacts violently with water, producing hydrochloric acid and heat
Ammonia gas	3/1/0	NA	15%/28%	Strong oxidizers, acids, halogens, salts of silver and zinc [Note: Corrosive to copper and galvanized surfaces]
4 Allyl 1,2 methylenedioxybenzene	NA	NA	NA/NA	NA
Ammonium chloride	2/0/0	NA	NA/NA	Acids, alkalis, and their associated carbonates; substance reacts with lead and silver salts to form a fulminating compound; substance reacts with ammonium compounds, bromine pentafluoride, bromine trifluoride, hydrogen cyanide, iodine heptafluoride, nitrates, and potassium chlorate
Ammonium formate	2/0/0	NA	NA/NA	Strong oxidizing agents
Ammonium hydroxide	3/1/0	NA	16%/27%	Acrolein; acrylic acid; chlorosulfonic acid; dimethyl sulfate; fluorine; gold + aqua regia; hydrochloric acid; hydrofluoric acid; iodine; nitric acid; oleum; propiolactone; propylene oxide; silver nitrate; silver oxide; silver oxide + ethyl alcohol; nitromethane; silver permanganate; sulfuric acid; halogens; forms explosive compounds with many heavy metals and halide salts
Aniline	3/2/0	158	1.3%/11%	Strong acids and strong oxidizers; albumin; solutions of iron, zinc, aluminum, toluene diisocyanate, and alkalis; ignites spontaneously in the presence of red fuming nitric acid and with sodium
Benzaldehyde	2/2/0	NA	1.4%/8.5%	Performic acid and other oxidizing materials; an explosion occurred after mixing sodium hydrosulfite, aluminum powder, potassium carbonate, and benzaldehyde
Benzene	2/3/0	12	1.2%/7.8%	Strong oxidizers, many fluorides and perchlorates, nitric acid

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Chemical	Hazards (NFPA Rating: H/F/R)	Flash Point (°F)	Explosive Limit (Lower/Upper)	Incompatibilities
Benzyl chloride	3/2/2	153	1.1%/14%	Oxidizers, acids, copper, aluminum, magnesium, iron, zinc, tin [Note: Can polymerize when in contact with all common metals except nickel and lead; hydrolyzes in H_2O to benzyl alcohol
Benzyl cyanide	2/1/0	223	NA/NA	Strong acids, strong bases, strong oxidizing agents, strong reducing agents, sodium hypochlorite
Bromobenzene	2/2/0	NA	0.5%/2.5%	Bromobutane + sodium, strong oxidizing agents, alkali metals
Bromoethane	3/1/0	NA	10%/16%	Risk of fire and explosion on contact with aluminum, zinc, or magnesium
Carbon dioxide	NA	NA	NA/NA	Dusts of various metals, such as magnesium, zirconium, titanium, aluminum, chromium, and manganese are ignitable and explosive when suspended in carbon dioxide; forms carbonic acid in water
Carbontetrachloride	3/0/0	NA	NA/NA	Aluminum, bromine trifluoride, calcium hypochlorite, dimethyl formamide, ethylene oxide, fluorine, lithium, magnesium, potassium, potassium-*tert*-butoxide, silver perchlorate, sodium, uranium, chlorine trifluoride, dinitrogen tetraoxide, methanol
Chloro-2-propanone	3/2/0	102	NA/NA	Strong acids, strong bases, strong oxidizing agents, strong reducing agents
Copper sulfate	2/0/0	NA	NA/NA	Moisture, air, steel, finely powdered metals, hydroxylamine, magnesium, hydrazine, nitromethane
Copper oxide	2/0/0	NA	NA/NA	Aluminum, boron, cesium acetylene carbide, hydrazine, magnesium, phospham, potassium, rubidium acetylene carbide, sodium, titanium, and zirconium; forms explosive acetylides with acetylene in caustic solutions; exposure to moist air at >212°F can result in spontaneous combustion
Cyclohexanone	1/2/0	111	1.1%/9.4%	Oxidizing agents, strong acids, amines, nitric acid, plastics, rubber, sulfuric acid, aliphatic amines, lead, red metals, resins

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Dichloroethane	2/4/2	>233	13%/23%	Strong oxidizing agents, liquid oxygen, nitric acid, potassium, lithium, sodium, caustics (e.g., ammonia, ammonium hydroxide, calcium hydroxide, potassium hydroxide, sodium hydroxide), potassium-*tert*-butoxide, sodium potassium alloys, powdered aluminum, active metals (such as potassium and magnesium), nitrogen tetroxide, *N*-methyl-*N*-nitososourea + potassium hydroxide, powdered magnesium
Ephedrine	1/0/0	NA	NA/NA	Oxidizing agents, direct light
Ethyl acetate	1/3/0	24	2.0%/9.0%	Chlorosulfonic acid, lithium aluminum hydride + 2-chloromethylfuran, lithium tetrahydroaluminate, oleum, potassium *t*-butoxide; substance coming in contact with nitrates or strong acids/oxidizers/alkalis may cause fire
Formamide	2/1/0	310	2.7%/19%	Strong oxidizing agents, acids, bases, aluminum
Formic acid	3/2/0	>233	18%/57%	Strong oxidizing agents, strong bases, finely powdered metals, permanganates, sulfuric acid, hydrogen peroxides, nitromethane, furfuryl alcohol, hydrated thallium nitrate
Hydrobromic acid (HBr)	3/0/0	NA	NA/NA	Strong oxidizers, strong caustics, moisture, copper, brass, zinc [Note: Hydrobromic acid is highly corrosive to most metals]
Hydrochloric acid (HCl)	3/0/0	NA	NA/NA	Hydroxides, amines, alkalis, copper, brass, zinc [Note: Hydrochloric acid is highly corrosive to most metals]
Hydrogen	0/4/0	Gas	4%/75%	Oxidizing agents, some metals, alkaline material, halogens
Hydrogen peroxide	4/0/1	NA	40%/100%	Strong oxidizing agents, strong reducing agents, acetic acid, acetic anhydride, alcohols, brass, copper, copper alloys, finely powdered metals, galvanized iron, hydrazine, iron, magnesium, nitric acid, sodium carbonate, potassium permanganate, cyanides (e.g., potassium cyanide, sodium cyanide), ethers [e.g., dioxane, furfuran, tetrahydrofuran (THF)], urea, chlorosulfonic acid, alkalis, lead, nitrogen compounds, triethylamine, silver, nickel, palladium, organic matter, charcoal, sodium borate, aniline, platinum, formic acid, cyclopentadiene, activated carbon, *tert*-butyl alcohol, hydrogen selenide, manganese dioxide, mercurous chloride, rust, ketones, carboxylic acids, glycerine, sodium fluoride, sodium pyrophosphate, soluble fuels (acetone, ethanol,glycerol), wood, asbestos, hexavalent chromium compounds, salts of iron, copper, chromium, vanadium, tungsten, molybdeum, and platinum [Note: Contact with combustible material may result in spontaneous combustion]

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Chemical	Hazards (NFPA Rating: H/F/R)	Flash Point (°F)	Explosive Limit (Lower/Upper)	Incompatibilities
Hydriodic acid (HI)	3/0/0	NA	NA/NA	Explodes on contact with ethyl hydroperoxide; ignites on contact with magnesium, perchloric acid, potassium + heat, potassium chlorate + heat, and oxidants; violent reaction with $HCLO_4$ + Mg, metals; potentially violent reaction with phosphorous
Hydroxylamine HCl	2/0/3	305	NA/NA	Strong oxidizing agents, heat plus sodium acetate or ether, carbonyl compounds, copper sulfate, zinc and phosphorus chlorides
Iodine	NA	NA	NA/NA	Incompatible with ammonia, powdered metals, alkali metals, or strong reducing agents; reaction can be violent or explosive with acetaldehyde and acetylene; reacts with ammonium hydroxide to form shock-sensitive iodides on drying
Isosafrole	1/1/0	120	NA/NA	Oxidizing agents
Lithium	3/2/2	NA	NA/NA	Moisture, acids, oxidizers, oxygen, nitrogen, carbon dioxide
Lithium aluminum hydroxide	3/0/2	NA	NA/NA	Water
Magnesium turnings	0/1/1	NA	NA/NA	Oxygen, moisture, chlorinated solvents, methanol, hydrogen peroxide, oxidizing agents, sulfur compounds, metal oxides, metal cyanides, metal oxide salts, fluorine, carbonates, halogens, phosphates
Manganous carbonate	1/0/1	NA	NA/NA	Contact with acids may generate carbon dioxide gas; oxidizes toxic sulfur dioxide to the more toxic sulfur trioxide and causes violent decomposition of hydrogen peroxide
Manganous chloride	1/0/1	NA	NA/NA	Strong reducing agents, hydrogen peroxide, potassium, sodium, and zinc
Mercuric chloride	4/0/1	NA	NA/NA	Reacts violently with potassium and sodium; incompatible with many compounds: formates, sulfites, phosphates, albumin, ammonia, gelatin, carbonates, hypophosphites, sulfides, alkalis, alkaloid salts, lime water, antimony and arsenic, bromides, borax, reduced iron, copper, iron, lead, tannic acid and vegetable astringents

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Mercury	3/0/0	NA	NA/NA	Acetylenes, ammonia, ethylene oxide, chlorine dioxide, azides, metal oxides, methyl silane, lithium, rubidium, oxygen, strong oxidants, metal carbonyls
Methanol	3/3/1	54	6%/36%	Strong oxidizing agents such as nitrates, perchlorates, or sulfuric acid; will attack some forms of plastics, rubber, and coatings; may react with metallic aluminum and generate hydrogen gas
Methylamine	3/3/0	39	4.9%/20.8%	Nitromethane, acids, oxidizing agents, chlorine, hypochlorite, halogenated agents, mercury, copper, copper alloys, zinc, zinc alloys, aluminum, perchlorates
Methylformamide	1/1/0	NA	NA/NA	Strong oxidizing agents, acids, bases, acid chlorides
Nitroethane	1/3/3	82	3.4%/NA	Amines; strong acids, alkalis, and oxidizers; hydrocarbons; combustibles; metal oxides
Norpseudoephedrine	1/0/0	NA	NA/NA	Oxidizing agents, direct light
Palladium sulfate	NA	NA	NA/NA	Strong oxidizing agents; protect from freezing
Perchloric acid	3/0/3	102	NA/NA	Incompatible with numerous materials, including combustible materials, organic chemicals, strong dehydrating agents, reducing and oxidizing agents; reacts violently with benzene, calcium hydride, wood, acetic acid, charcoal, olefins, ethanol, sulfur, and sulfuric acid; do not use perchloric acid in a hood designed for other purposes
Phenylacetic acid	2/0/0	168	NA/NA	NA
Phenylmagnesium bromide	NA	NA	NA/NA	Water
Phenylacetone	NA	NA	NA/NA	NA
Phosphorus (red)	0/2/2	NA	NA/NA	Halogens, halides, sulfur, oxidizing materials, and alkalis (forms phosphine)
Phosphorus pentachloride	3/0/2	NA	NA/NA	Reacts violently with water; alcohols, amines, aluminum, sodium, potassium acids
Piperonal	1/0/0	NA	NA/NA	Strong oxidizing agents
Piperidine	3/3/0	NA	NA/NA	Acids, acid chlorides, acid anhydrides, carbon dioxide, strong oxidizing agents, dicyanofurazan, *N*-nitrosoacetanilide, 1-perchlorylpiperdine

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Chemical	Hazards (NFPA Rating: H/F/R)	Flash Point (°F)	Explosive Limit (Lower/Upper)	Incompatibilities
Platinum	NA	NA	NA/NA	Aluminum, acetone, arsenic, ethane, hydrazine, hydrogen peroxide, lithium, phosphorus, selenium, tellurium, various fluorides
Platinum chloride	NA	1076	NA/NA	Strong oxidizing agents
Platinum oxide	2/0/0	NA	NA/NA	Oxidizing agents
Potassium carbonate	2/0/1	NA	NA/NA	Acids, chlorine trifluoride, magnesium; an explosion occurred after mixing sodium hydrosulfite, aluminum powder, potassium carbonate, and benzaldehyde
Potassium cyanide	4/0/0	NA	NA/NA	Violent reactions can occur with oxidizing agents such as nitric acid, nitrates, and peroxides; contact with acids liberates extremely toxic and flammable hydrogen cyanide gas; hydrogen cyanide may form by a reaction with carbon dioxide and moisture when this material is in prolonged contact with air in a closed system
Potassium hydroxide	3/0/2	NA	NA/NA	Contact with water, acids, flammable liquids, and organic halogen compounds, especially trichloroethylene, may cause fire or explosion; contact with nitromethane and other similar nitro compounds causes formation of shock-sensitive salts; contact with metals such as aluminum, tin, and zinc causes formation of flammable hydrogen gas
Pumice	NA	NA	NA/NA	NA
Pyridine	2/3/0	68	1.8%/12.4%	Strong oxidizers, strong acids
Raney nickel	NA	NA	NA/NA	NA
Sodium	3/3/3	NA	NA/NA	Water, oxygen, carbon dioxide, carbon tetrachloride, halogens, acetylene, metal halides, ammonium salts, oxides, oxidizing agents, acids, alcohols, chlorinated organic compounds, many other substances

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Sodium acetate	3/0/1	NA	NA/NA	Nitric acid, fluoride, potassium nitrate, strong oxidizers, and diketene
Sodium bisulfate	IRR	NA	NA/NA	Strong bases, strong oxidizing agents, strong reducing agents
Sodium hydroxide	3/0/2	NA	NA/NA	Metals, acids, nitro compounds, halogenated organics (e.g., dibromoethane, hexachlorobenzene, methyl chloride, trichloroethylene), nitromethane, flammable liquids
Sodium sulfate	1/0/0	NA	NA/NA	Strong oxidizing agents, aluminum, magnesium, potassium, mercury, lead, calcium, silver, barium, ammonium ions, strontium
Sulfuric acid	3/0/3	NA	NA/NA	Water, potassium chlorate, potassium perchlorate, potassium permanganate, sodium, lithium, bases, organic material, halogens, metal acetylides, oxides and hydrides, metals (yields hydrogen gas), strong oxidizing and reducing agents, and many other reactive substances
Thionyl chloride	4/0/2	NA	NA/NA	Water, ammonia, chloryl perchlorate, dimethyl sulfoxide, linseed oil, quinoline, sodium, 2,4-hexadiyn-1–6-diol, *o*-nitrobenzoyl acetic acid, and *o*-nitrophenylacetic acid
Thorium nitrate	NA	NA	NA/NA	Strong oxidizers

Continued.

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Key to NFPA ratings

Health (H) Hazard — The Left Quadrant (Blue)	Fire (F) Hazard — The Top Quadrant (Red)	Reactivity (R) Hazard — The Right Quadrant (Yellow)	Important Messages — The Bottom Quadrant (White)
4. DANGER: May be fatal on short exposure. Specialized protective equipment required.	4. DANGER: Flammable gas or extremely flammable liquid.	4. DANGER: Explosive material at room temperature.	W — Avoid use of water COR — Corrosive LAS — Laser electrical hazard AZK — Alkali
3. WARNING: Corrosive or toxic. Avoid skin contact or inhalation.	3. WARNING: Flammable liquid. Flash point below 100°F.	3. DANGER: May be explosive if shocked, heated under confinement, or mixed with water.	ACID — Acid OXY — Oxidizing chemicals RED — Dangerous reducing agent/metal hydride
2. WARNING: May be harmful if inhaled or absorbed.	2. CAUTION: Combustible liquid. Flash point of 100 to 200°F.	2. WARNING: Unstable, or may react if heated or if mixed with water.	
1. CAUTION: May cause irritation.	1. Combustible if heated.	1. CAUTION: May react if heated or mixed with water.	
0. No unusual hazard.	0. Not combustible.	0. Stable. Not reactive when mixed with water.	

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Appendix F: Toxicology Table

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Chemical	Effects	Target Organs	IDLH (ppm)	NIOSH REL (ppm)	OSHA PEL (ppm)
Acetaldehyde	Causes severe eye irritation; vapors may cause eye irritation; may cause transient corneal injury or lachrymator (substance which increases the flow of tears); may cause skin irritation; may cause skin sensitization, an allergic reaction, which becomes evident upon reexposure to this material; may cause gastrointestinal irritation with nausea, vomiting, and diarrhea; may be harmful if swallowed; causes respiratory tract irritation; may cause narcotic effects in high concentrations; exposure produces central nervous system depression; vapors may cause dizziness or suffocation; can produce delayed pulmonary edema; inhalation of large amounts may cause respiratory stimulation, followed by respiratory depression, convulsions, and possible death due to respiratory paralysis	Eyes, skin, respiratory system, kidneys, central nervous system, reproductive system	2000		200
Acetic acid	Causes severe eye irritation; contact with liquid or vapor causes severe burns and possible irreversible eye damage; causes skin burns; may be harmful if absorbed through the skin; contact with the skin may cause blackening and hyperkeratosis of the skin of the hands; may cause severe and permanent damage to the digestive tract; causes severe pain, nausea, vomiting, diarrhea, and shock; may cause polyuria, oliguria, and anuria; rapidly absorbed from the gastrointestinal tract; effects may be delayed; causes chemical burns to the respiratory tract; exposure may lead to bronchitis, pharyngitis, and dental erosion; may be absorbed through the lungs	Eyes, skin, respiratory system, teeth	50	10	10
Acetic anhydride	In case of contact with eyes — immediately flush eyes with plenty of water for at least 15 min, and get medical aid immediately; in case of contact with skin — immediately flush skin with plenty of water for at least 15 min, while removing contaminated clothing and shoes, and get medical aid immediately, wash clothing before reuse; if swallowed — do NOT induce vomiting, get medical aid immediately, give a cupful of water to victim if fully conscious (never give anything by mouth to an unconscious person); if inhaled — remove to fresh air, give artificial respiration if victim is not breathing, give oxygen if breathing is difficult, and get medical aid	Central nervous system, eyes, skin, mucous membranes	200	5	5

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Acetone	Flush eyes with plenty of water for at least 15 min, occasionally lifting the upper and lower eyelids, and get medical aid immediately; flush skin with plenty of soap and water for at least 15 min, while removing contaminated clothing and shoes, and get medical aid if irritation develops or persists, wash clothing before reuse; If swallowed — do NOT induce vomiting, give two to four cupfuls of milk or water to victim if conscious and alert (never give anything by mouth to an unconscious person), get medical aid immediately; If inhaled — remove from exposure to fresh air immediately, give artificial respiration if victim is not breathing, give oxygen if breathing is difficult, get medical aid immediately, do NOT use mouth-to-mouth resuscitation, apply artificial respiration using oxygen and a suitable mechanical device such as a bag and a mask if breathing has ceased	Eyes, skin, respiratory system, central nervous system	2500	250	1000
Acetonitrile	Causes eye irritation; lachrymator (substance which increases the flow of tears); may produce superficial reversible injury; causes mild skin irritation; harmful if absorbed through the skin; may be metabolized to cyanide which in turn acts by inhibiting cytochrome oxidase and impairing cellular respiration; may cause gastrointestinal irritation with nausea, vomiting, and diarrhea; may cause effects similar to those for inhalation exposure; may cause tissue anoxia, characterized by weakness, headache, dizziness, confusion, cyanosis (bluish skin due to deficient oxygenation of the blood), weak and irregular heartbeat, collapse, unconsciousness, convulsions, coma, and death; may cause central nervous system depression; metabolism may release cyanide, which may result in headache, dizziness, weakness, collapse, unconsciousness, and possible death; aspiration may lead to pulmonary edema; vapors may cause dizziness or suffocation; causes upper respiratory tract irritation	Respiratory system, cardiovascular system, central nervous system, liver, kidneys	500	20	40
Allylbenzene	Contact with eyes — immediately flush eyes with plenty of water for at least 15 min, occasionally lifting the upper and lower eyelids, and get medical aid; Skin contact — flush skin with plenty of soap and water for at least 15 min while removing contaminated clothing and shoes, get medical aid if irritation develops or persists, wash clothing before reuse; If swallowed — give two to four cupfuls of milk or water to victim if conscious and alert (never give anything by mouth to an unconscious person), and get medical aid; If inhaled — remove from exposure to fresh air immediately, give artificial respiration if victim is not breathing, give oxygen if breathing is difficult, and get medical aid if cough or other symptoms appear	None	None listed	None listed	None listed

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Chemical	Effects	Target Organs	IDLH (ppm)	NIOSH REL (ppm)	OSHA PEL (ppm)
Allylchloride	Irritation of eyes, skin, nose, mucous membranes; pulmonary edema; in animals: liver, kidney injury	Eyes, skin, respiratory system, liver, kidneys	250	1	1
4 Allyl 1,2 methylenedioxy-benzene	None listed	None listed	None listed	None listed	None listed
Aluminum chloride	Causes severe eye burns; causes skin burns; causes gastrointestinal tract burns; may cause corrosion and permanent tissue destruction of the esophagus and digestive tract; causes delayed lung injury; causes severe irritation of upper respiratory tract with coughing, burns, breathing difficulty, and possible coma	Eyes, skin, mucous membranes		10 mg/m^3	15 mg/m^3
Ammonia gas	Irritation of eyes, nose, throat; dyspnea (breathing difficulty), wheezing, chest pain; pulmonary edema; pink frothy sputum; skin burns, vesiculation; liquid: frostbite	Eyes, skin, respiratory system	300	25	50
Ammonium chloride	Irritation of eyes, skin, respiratory system; cough, dyspnea (breathing difficulty), pulmonary sensitization	Eyes, skin, respiratory system		10 mg/m^3	none
Ammonium formate	Causes eye irritation; causes skin irritation; may cause gastrointestinal irritation with nausea, vomiting, and diarrhea; toxicological properties of this substance have not been fully investigated; may cause respiratory tract irritation	None listed	None listed	None listed	None listed
Ammonium hydroxide	Contact with liquid or vapor causes severe burns and possible irreversible eye damage; causes severe skin irritation; causes skin burns; may cause deep, penetrating ulcers of the skin; contact with the skin may cause staining, inflammation, and thickening of the skin; harmful if swallowed; may cause severe and permanent damage to the digestive tract; causes gastrointestinal tract burns; causes throat constriction, vomiting, convulsions, and shock; effects may be delayed; causes severe irritation of upper respiratory tract with coughing, burns, breathing difficulty, and possible coma	Eyes, skin, mucous membranes	None listed	None listed	None listed
Aniline	Headache, lassitude (weakness, exhaustion), dizziness; cyanosis; ataxia; dyspnea (breathing difficulty) on effort; tachycardia; irritation of eyes; methemoglobinemia; cirrhosis; [potential occupational carcinogen]	Blood, cardiovascular system, eyes, liver, kidneys, respiratory system	100	None listed	5

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Benzaldehyde	Causes eye irritation; causes skin irritation; harmful if swallowed; may cause gastrointestinal irritation with nausea, vomiting, and diarrhea; may cause central nervous system depression, characterized by excitement, followed by headache, dizziness, drowsiness, and nausea; advanced stages may cause collapse, unconsciousness, coma, and possible death due to respiratory failure; inhalation of high concentrations may cause central nervous system effects characterized by nausea, headache, dizziness, unconsciousness, and coma; may cause respiratory tract irritation; may cause narcotic effects in high concentrations; prolonged or repeated skin contact may cause dermatitis; may cause kidney injury	Kidneys, central nervous system	None listed	None listed	None listed
Benzene	Irritation of eyes, skin, nose, respiratory system; dizziness; headache, nausea, staggered gait; anorexia, lassitude (weakness, exhaustion); dermatitis; bone marrow depression; [potential occupational carcinogen]	Eyes, skin, respiratory system, blood, central nervous system, bone marrow Cancer Site [leukemia]	500	0.1	1
Benzyl chloride	Irritation of eyes, skin, nose; lassitude (weakness, exhaustion); irritability; headache; skin eruption; pulmonary edema	Eyes, skin, respiratory system, central nervous system	10	1	1
Benzyl cyanide	Causes eye irritation; causes skin irritation; harmful if absorbed through the skin; may be metabolized to cyanide which, in turn, acts by inhibiting cytochrome oxidase and impairing cellular respiration; harmful if swallowed; may cause irritation of the digestive tract; metabolism may release cyanide, which may result in headache, dizziness, weakness, collapse, unconsciousness, and possible death; ingestion may result in symptoms similar to cyanide poisoning, which is characterized by asphyxiation; may be fatal if inhaled; may cause effects similar to those described for ingestion	Blood, kidneys, liver, spleen, brain	Not listed	Not listed	5 mg/m^3
Bromobenzene	Causes eye irritation; causes skin irritation; may be absorbed through the skin in harmful amounts; if absorbed, may cause liver injury; causes gastrointestinal irritation with nausea, vomiting, and diarrhea; may cause central nervous system depression; inhalation of high concentrations may cause central nervous system effects characterized by nausea, headache, dizziness, unconsciousness, and coma; causes respiratory tract irritation; may cause narcotic effects in high concentrations; may cause liver abnormalities; vapors may cause dizziness or suffocation; may cause blood changes	Blood, central nervous system, liver	Not listed	Not listed	Not listed

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Chemical	Effects	Target Organs	IDLH (ppm)	NIOSH REL (ppm)	OSHA PEL (ppm)
Bromoethane	Irritates the eyes, the skin, and the respiratory tract; inhalation may cause lung edema; rapid evaporation of the liquid may cause frostbite; may cause effects on the central nervous system, kidneys, and lungs; exposure to high concentrations may result in death; effects may be delayed	Central nervous system, kidneys, lungs	Not listed	Not listed	Not listed
Carbon dioxide	Headache, dizziness, restlessness, paresthesia; dyspnea (breathing difficulty); sweating, malaise (vague feeling of discomfort); increased heart rate, cardiac output, blood pressure; coma; asphyxia; convulsions; frostbite (liquid, dry ice)	Respiratory system, cardiovascular system	40,000	5000	5000
Carbon tetrachloride	Irritation of eyes, skin; central nervous system depression; nausea, vomiting; liver, kidney injury; drowsiness, dizziness, uncoordination; [potential occupational carcinogen]	Central nervous system, eyes, lungs, liver, kidneys, skin	200	2	10
Chloro-2-propanone (chloroacetone)	Causes eye burns; lachrymator (substance which increases the flow of tears); may cause chemical conjunctivitis and corneal damage; may be fatal if absorbed through the skin; causes skin burns; may cause cyanosis of the extremities; may cause skin rash (in milder cases), and cold and clammy skin with cyanosis or pale color; harmful if swallowed; may cause severe and permanent damage to the digestive tract; may cause gastrointestinal irritation with nausea, vomiting, and diarrhea; may cause liver and kidney damage; may cause perforation of the digestive tract; ingestion of large amounts may cause CNS depression; may cause spleen damage; may cause systemic effects; may be fatal if inhaled; causes chemical burns to the respiratory tract; aspiration may lead to pulmonary edema; vapors may cause dizziness or suffocation; may cause systemic effects; vapors are extremely irritating to the respiratory tract; may cause burning sensation in the chest	Kidneys, central nervous system, liver, spleen	None listed	None listed	None listed

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Copper sulfate	Exposure to particulates or solution may cause conjunctivitis, ulceration, and corneal abnormalities; causes eye irritation and possible burns; may cause skin sensitization, an allergic reaction, which becomes evident upon reexposure to this material; causes skin irritation and possible burns; may cause eczema; harmful if swallowed; may cause severe gastrointestinal tract irritation with nausea, vomiting, and possible burns; ingestion of large amounts of copper salts may cause bloody stools and vomit, low blood pressure, jaundice, and coma; ingestion of copper compounds may produce systemic toxic effects to the kidney and liver and central nervous excitation followed by depression; may cause ulceration and perforation of the nasal septum if inhaled in excessive quantities; causes respiratory tract irritation with possible burns	Blood, kidneys, liver	100 mg/m^3	1 mg/m^3	1 mg/m^3
Copper oxide	Causes eye irritation; may result in corneal injury; may cause conjunctivitis; causes skin irritation; may cause skin discoloration; may cause central nervous system depression, kidney damage, and liver damage; may cause gastrointestinal irritation with nausea, vomiting, and diarrhea; may cause circulatory system failure; may cause vascular collapse and damage; causes respiratory tract irritation; may cause ulceration and perforation of the nasal septum if inhaled in excessive quantities; inhalation of fumes may cause metal fume fever, which is characterized by flu-like symptoms with metallic taste, fever, chills, cough, weakness, chest pain, muscle pain, and increased white blood cell count	Kidneys, central nervous system, liver, red blood cells	100 mg/m^3	1 mg/m^3	1 mg/m^3
Cyclohexanone	May result in corneal injury; vapors may cause eye irritation; contact produces irritation, tearing, and burning pain; causes skin irritation; harmful if absorbed through the skin; causes gastrointestinal irritation with nausea, vomiting, and diarrhea; may cause liver and kidney damage; may cause central nervous system depression, characterized by excitement, followed by headache, dizziness, drowsiness, and nausea — advanced stages may cause collapse, unconsciousness, coma, and possible death due to respiratory failure; may be harmful if swallowed; inhalation of high concentrations may cause central nervous system effects characterized by nausea, headache, dizziness, unconsciousness, and coma; may cause liver and kidney damage; may cause narcotic effects in high concentrations; inhalation may be fatal as a result of spasm, inflammation, edema of the larynx and bronchi, chemical pneumonitis, and pulmonary edema; may cause irritation of the mucous membranes	Eyes, skin, respiratory system, central nervous system, liver, kidneys	700	25	50

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Chemical	Effects	Target Organs	IDLH (ppm)	NIOSH REL (ppm)	OSHA PEL (ppm)
Dichloroethane	Contact with eyes may cause severe irritation and possible eye burns; may be absorbed through the skin; causes irritation with burning pain, itching, and redness; prolonged exposure may result in skin burns; causes gastrointestinal irritation with nausea, vomiting, and diarrhea; may cause central nervous system depression, characterized by excitement, followed by headache, dizziness, drowsiness, and nausea — advanced stages may cause collapse, unconsciousness, coma, and possible death due to respiratory failure; may be harmful if swallowed; inhalation of high concentrations may cause central nervous system effects characterized by nausea, headache, dizziness, unconsciousness, and coma; causes respiratory tract irritation; may cause narcotic effects in high concentrations; vapors may cause dizziness or suffocation; may cause blood changes; overexposure may cause an increase in carboxyhemoglobin levels in the blood; can produce delayed pulmonary edema	Blood, heart, central nervous system, liver, pancreas	3000	100	100
Ephedrine	May cause eye irritation; may cause skin irritation; contact with the skin may cause a local anesthetic effect; may cause irritation of the digestive tract; may cause respiratory tract irritation; toxicological properties of this substance have not been fully investigated	Heart, nerves	None listed	None listed	None listed
Ethyl acetate	Causes eye irritation; vapors may cause eye irritation; may cause skin irritation; prolonged or repeated contact may cause irritation and dermatitis; may cause irritation of the digestive tract; may cause liver and kidney damage; ingestion of large amounts may cause central nervous depression; may cause headache, nausea, fatigue, and dizziness; may cause respiratory tract irritation; may be harmful if inhaled; inhalation of high concentrations may cause narcotic effects	Kidneys, central nervous system, liver	2000	400	400
Formamide	Causes eye irritation; causes skin irritation; may cause irritation of the digestive tract; inhalation of high concentrations may cause central nervous system effects characterized by nausea, headache, dizziness, unconsciousness, and coma; may cause respiratory tract irritation	Central nervous system	None listed	10	None listed

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Formic acid	Contact with liquid is corrosive to the eyes and causes severe burns; lachrymator (substance which increases the flow of tears); may cause corneal edema, ulceration, and scarring; may cause skin sensitization, an allergic reaction, which becomes evident upon reexposure to this material; contact with liquid is corrosive and causes severe burns and ulceration; it is absorbed through the skin; may cause erythema (redness) and blistering; causes severe digestive tract burns with abdominal pain, vomiting, and possible death; may be harmful if swallowed; may cause central nervous system depression; ingestion may produce corrosive ulceration and bleeding and necrosis of the gastrointestinal tract accompanied by shock and circulatory collapse; may cause asthmatic attacks due to allergic sensitization of the respiratory tract; causes chemical burns to the respiratory tract; aspiration may lead to pulmonary edema; vapors may cause dizziness, nausea, itching, burning, and swelling of the eyes	Kidneys, central nervous system, liver, respiratory system, eyes, skin	30	5	5
Hydrobromic acid (HBr)	Eye contact may result in corneal injury; causes severe eye irritation and burns; causes severe skin irritation; may be absorbed through the skin; contact with liquid is corrosive and causes severe burns and ulcerations; causes gastrointestinal tract burns; may cause respiratory failure; may cause circulatory system failure; may cause hemorrhaging of the digestive tract; may cause corrosion and permanent tissue destruction of the esophagus and digestive tract; irritation may lead to chemical pneumonitis and pulmonary edema; causes chemical burns to the respiratory tract; may cause effects similar to those described for ingestion	None	30	3	3
Hydrochloric acid (HCl)	May cause irreversible eye injury; vapor or mist may cause irritation and severe burns; contact with liquid is corrosive to the eyes and causes severe burns; may cause painful sensitization to light; may be absorbed through the skin in harmful amounts; may cause skin sensitization, an allergic reaction, which becomes evident upon reexposure to this material; contact with liquid is corrosive and causes severe burns and ulcerations; may cause circulatory system failure; causes severe digestive tract burns with abdominal pain, vomiting, and possible death; may cause corrosion and permanent tissue destruction of the esophagus and digestive tract; may be harmful if swallowed; may cause severe irritation of the respiratory tract with sore throat, coughing, shortness of breath, and delayed lung edema; causes chemical burns to the respiratory tract; exposure to the mist and vapor may erode exposed teeth; causes corrosive action on the mucous membranes	Toxic by inhalation; causes severe burns; corrosive; mutagen	50	5	5

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Chemical	Effects	Target Organs	IDLH (ppm)	NIOSH REL (ppm)	OSHA PEL (ppm)
Hydrogen	Defined as a simple asphyxiant; inhalation of high concentrations of hydrogen may cause dizziness, headache, deeper breathing due to air hunger, possible nausea, and eventual unconsciousness; eyes/skin/oral: not likely to occur	Lungs and central nervous system			
Hydrogen peroxide	Contact with liquid is corrosive to the eyes and causes severe burns; contact with the eyes may cause corneal damage; causes severe skin irritation and possible burns; may cause discoloration, erythema (redness), swelling, and the formation of papules and vesicles (blisters); causes gastrointestinal irritation with nausea, vomiting, and diarrhea; causes gastrointestinal tract burns; may cause vascular collapse and damage; may cause damage to the red blood cells; may cause difficulty in swallowing, stomach distension, possible cerebral swelling, and death; ingestion may result in irritation of the esophagus, bleeding of the stomach, and ulcer formation; causes chemical burns to the respiratory tract; may cause ulceration of nasal tissue, insomnia, nervous tremors with numb extremities, chemical pneumonia, unconsciousness, and death; at high concentrations, respiratory effects may include acute lung damage and delayed pulmonary edema	Blood, central nervous system	75	1	1
Hydriodic acid (HI)	Causes eye burns; causes skin burns; may cause severe and permanent damage to the digestive tract; causes gastrointestinal tract burns; may cause irritation of the respiratory tract with burning pain in the nose and throat, coughing, wheezing, shortness of breath, and pulmonary edema; causes chemical burns to the respiratory tract; inhalation may be fatal as a result of spasm, inflammation, edema of the larynx and bronchi, chemical pneumonitis, and pulmonary edema	None known	None Listed	None Listed	None Listed

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Hydroxylamine HCl	Corrosive; extremely destructive to tissues of the mucous membranes and upper respiratory tract; symptoms may include burning sensation, coughing, wheezing, laryngitis, shortness of breath, headache, nausea, and vomiting; inhalation may be fatal as a result of spasm inflammation and edema of the larynx and bronchi, chemical pneumonitis, and pulmonary edema; may convert hemoglobin to methemoglobin, producing cyanosis; may also cause nausea, vomiting, drop in blood pressure, headache, vertigo, ringing in the ears, shortness of breath, severe blood oxygen deficiency, and convulsions; high concentrations cause coma and death from circulatory collapse; irritant and possible sensitizer; may cause burns; corrosive to the eyes; may cause severe irritation and corneal damage	Kidneys, central nervous system, eyes, blood, skin, liver, or lungs	None Listed	None Listed	None Listed
Iodine	Causes severe eye irritation; may cause eye burns; vapor or mist may cause irritation and severe burns; causes skin burns; may cause skin sensitization, an allergic reaction, which becomes evident upon reexposure to this material; may cause gastrointestinal irritation with nausea, vomiting, and diarrhea; may cause kidney damage; may cause burns to the digestive tract; may cause thyroid abnormalities; may cause irritation of the respiratory tract with burning pain in the nose and throat, coughing, wheezing, shortness of breath, and pulmonary edema; may cause epiphoria, which is an excessive flow of tears	Kidneys, thyroid	None Listed	2	0.1
Isosafrole	May cause eye irritation; may cause skin irritation; may cause irritation of the digestive tract; may cause liver damage; may cause cyanosis (bluish discoloration of the skin due to deficient oxygenation of the blood), weakness, acidosis, and shock; may be harmful if swallowed; may cause respiratory tract irritation; may cause effects similar to those described for ingestion	Liver	None Listed	None Listed	None Listed
Lithium	There is no known long-term hazard from lithium in its solid state — however, lithium metal is extremely reactive with body moisture and is corrosive to the skin, nose, throat, and eyes	None	None Listed	None Listed	None Listed

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Chemical	Effects	Target Organs	IDLH (ppm)	NIOSH REL (ppm)	OSHA PEL (ppm)
Lithium aluminum hydroxide	Causes eye burns; causes irritation when substance becomes wet or comes in contact with moisture of the mucous membranes; may cause chemical conjunctivitis and corneal damage; causes skin burns; contact with skin causes irritation and possible burns, especially if the skin is wet or moist; may cause skin rash (in milder cases), and cold and clammy skin with cyanosis or pale color; may cause severe and permanent damage to the digestive tract; causes gastrointestinal tract burns; may cause perforation of the digestive tract; may cause systemic effects; causes chemical burns to the respiratory tract; aspiration may lead to pulmonary edema; may cause systemic effects	None	None Listed	15 mg/m^3	10 mg/m^3
Magnesium turnings	Dust may cause mechanical irritation; may cause skin irritation; particles embedded in the skin may cause "chemical gas gangrene" with symptoms of persistent lesions, inflammation, and gas bubbles under the skin; may cause irritation of the digestive tract; may cause respiratory tract irritation; inhalation of fumes may cause metal fume fever, which is characterized by flu-like symptoms with metallic taste, fever, chills, cough, weakness, chest pain, muscle pain, and increased white blood cell count	None	None Listed	None Listed	None Listed
Manganous carbonate	Acute poisoning can occur from excessive inhalation causing symptoms noted under Chronic Exposure; extremely large oral dosages may produce gastrointestinal disturbances and acute poisoning, as noted under Chronic Exposure; no adverse effects are expected with dermal exposure; in the case of eye contact, no adverse effects are expected, but dust may cause mechanical irritation	None	None Listed	None Listed	5 mg/m^3
Manganous chloride	Inhalation can cause a flu-like 24 h to 48 h illness (metal fume fever) characterized by chills, fever, aching muscles, dryness in the mouth and throat, and headache; may irritate the respiratory tract; may increase the incidence of upper respiratory infections (pneumonia); absorption of inorganic manganese salts through the lungs is poor but may occur in chronic poisoning; ingestion — may cause abdominal pain and nausea, and although poorly absorbed through the intestines, inorganic manganese salts may produce hypoglycemia and decreased calcium blood levels should absorption occur; may cause irritation with redness and pain	Brain, kidney, blood			

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Mercuric chloride	Inhalation — causes irritation to the respiratory tract, with symptoms including sore throat, coughing, pain, tightness in chest, breathing difficulties, shortness of breath, and headache; pneumonitis may develop; can be absorbed through inhalation, with symptoms similar to those if ingested; vapor inhalation can burn the mucous membranes of the nose and throat; Ingestion — Highly Toxic! with an average lethal dose of about 1 g for inorganic mercury salts; may cause burning of the mouth and pharynx, abdominal pain, vomiting, corrosive ulceration, bloody diarrhea which may be followed by a rapid and weak pulse, shallow breathing, paleness, exhaustion, central nervous system problems, tremors, and collapse; delayed death may occur from renal failure; causes irritaton and burns to skin with symptoms including redness and pain; may cause skin allergy and sensitization; can be absorbed through the skin with symptoms to parallel ingestion; causes irritation and burns to eyes with symptoms including redness, pain, blurred vision; may cause serious and permanent eye damage	Eyes, skin, respiratory system, central nervous system, kidneys	10 mg/m^3	0.05 mg/m^3	0.1 mg/m^3
Mercury	Eye exposure — exposure to mercury or mercury compounds can cause discoloration on the front surface of the lens that does not interfere with vision, causes eye irritation and possible burns, contact with mercury or mercury compounds can cause ulceration of the conjunctiva and cornea; Dermal contact — may be absorbed through the skin in harmful amounts, may cause skin sensitization (an allergic reaction that becomes evident upon reexposure to this material), causes skin irritation and possible burns, may cause skin rash (in milder cases), and may cause cold and clammy skin with cyanosis or pale color; may cause severe and permanent damage to the digestive tract, may cause perforation of the digestive tract; may cause effects similar to those for inhalation exposure; may cause systemic effects; causes chemical burns to the respiratory tract; inhalation of fumes may cause metal fume fever, which is characterized by flu-like symptoms with metallic taste, fever, chills, cough, weakness, chest pain, muscle pain, and increased white blood cell count; may cause central nervous system effects including vertigo, anxiety, depression, muscle uncoordination, and emotional instability; aspiration may lead to pulmonary edema; may cause systemic effects; may cause respiratory sensitization	Blood, kidneys, central nervous system, liver, brain	10 mg/m^3	0.05 mg/m^3	0.1 mg/m^3

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Chemical	Effects	Target Organs	IDLH (ppm)	NIOSH REL (ppm)	OSHA PEL (ppm)
Methanol	Causes moderate eye irritation; vapors may cause eye irritation; may cause painful sensitization to light; may cause skin irritation; may be absorbed through the skin; may be fatal or cause blindness if swallowed; may cause irritation of the digestive tract; may cause kidney damage; may cause central nervous system depression, characterized by excitement, followed by headache, dizziness, drowsiness, and nausea — advanced stages may cause collapse, unconsciousness, coma, and possible death due to respiratory failure; may cause respiratory tract irritation; may cause adverse central nervous system effects including headache, convulsions, and possible death; may cause visual impairment and possible permanent blindness; may cause effects similar to those described for ingestion; may cause kidney damage	Kidneys, central nervous system, eyes	6000	200	200
Methylamine	Causes eye burns; may result in corneal injury; may cause chemical conjunctivitis and corneal damage; may cause tearing, conjunctivitis, and corneal edema when vapor is absorbed into the tissue of the eye; causes skin burns; may be absorbed through the skin; may cause dermatitis; methylamine is readily absorbed through the skin and may cause malaise, discomfort, injury, and death unless treated promptly; harmful if swallowed; causes gastrointestinal tract burns; causes chemical burns to the respiratory tract; may cause pulmonary edema and severe respiratory disturbances; may cause liver abnormalities; inhalation of methylamine may cause coughing, nausea, and pulmonary edema; in an unpublished report, allergic or chemical bronchitis was reported in a worker exposed to methylamine (actual exposure concentrations were not reported)	Liver, respiratory system, eyes, skin	100	10	10
Methylformamide	Causes eye irritation; may cause chemical conjunctivitis; causes skin irritation; may cause gastrointestinal irritation with nausea, vomiting, and diarrhea; causes respiratory tract irritation; can produce delayed pulmonary edema	Reproductive system	None listed	None listed	None listed

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Nitroethane	Causes eye irritation; may cause chemical conjunctivitis and corneal damage; causes skin irritation; may cause dermatitis; may cause cyanosis of the extremities; may cause gastrointestinal irritation with nausea, vomiting, and diarrhea; methemoglobinemia is characterized by dizziness, drowsiness, headache, shortness of breath, cyanosis with bluish skin, rapid heart rate, and chocolate-brown colored blood; ingestion of large amounts may cause CNS depression; may be harmful if swallowed; may form methemoglobin which in sufficient concentrations causes cyanosis (bluish discoloration of skin due to deficient oxygenation of the blood); causes respiratory tract irritation; aspiration may lead to pulmonary edema; vapors may cause dizziness or suffocation; may cause burning sensation in the chest	Kidneys, central nervous system, liver, respiratory system, skin	1000	100	100
Norpseudoephedrine	May cause eye irritation; may cause skin irritation; contact with the skin may cause a local anesthetic effect; may cause irritation of the digestive tract; may cause respiratory tract irritation; toxicological properties of this substance have not been fully investigated	Heart, nerves	None listed	None listed	None listed
Palladium sulfate	May be harmful by inhalation, ingestion, or skin absorption; may cause eye and skin irritation; to the best of the manufacturer's knowledge, the toxicological properties have not been thoroughly investigated	None listed	None listed	None listed	None listed
Perchloric acid	Causes eye burns; may cause retinal damage; causes skin burns; may cause skin sensitization, an allergic reaction that becomes evident upon reexposure to this material; may cause deep, penetrating ulcers of the skin; causes gastrointestinal tract burns; may be harmful if swallowed; ingestion may produce corrosive ulceration and bleeding and necrosis of the gastrointestinal tract accompanied by shock and circulatory collapse; may cause severe irritation of the respiratory tract, with sore throat, coughing, shortness of breath, and delayed lung edema; inhalation may be fatal as a result of spasm, inflammation, edema of the larynx and bronchi, chemical pneumonitis, and pulmonary edema	Eyes, skin, mucous membranes	None Listed	None Listed	None Listed
Phenylacetic acid	Causes eye irritation and possible burns; may cause chemical conjunctivitis; causes skin irritation and possible burns; may cause gastrointestinal irritation with nausea, vomiting, and diarrhea; causes respiratory tract irritation; can produce delayed pulmonary edema	None listed	None listed	None listed	None listed

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Chemical	Effects	Target Organs	IDLH (ppm)	NIOSH REL (ppm)	OSHA PEL (ppm)
Phenylmagnesium bromide	NA	NA	NA	NA	NA
Phenylacetone	NA	NA	NA	NA	NA
Phosphorus	Not considered highly toxic, but acute exposure may cause coughing, bronchitis, possible liver or kidney impairment if contaminated with yellow phosphorus; red phosphorus is not readily absorbed and, in pure form, is considered nonpoisonous, however, possible contamination with the yellow form must be considered, and symptoms such as nausea, vomiting, abdominal pain, or garlic odor on breath will indicate poisoning by the latter — estimated lethal adult human dose for white phosphorus is 50 to 100 mg; red phosphorous is not harmful to skin; if contaminated with white phosphorus, however, contact may cause deep slow-healing burns; red phosphorus causes eye irritation; if contaminated with yellow phosphorus, eye contact can cause severe irritation and burns	Kidneys, liver	5 mg/m^3	0.1 mg/m^3	0.1 mg/m^3
Phosphorus pentachloride	Causes severe eye burns; causes skin burns; causes digestive tract burns with immediate pain, swelling of the throat, convulsions, and possible coma; may be harmful if swallowed; may cause severe irritation of the respiratory tract with sore throat, coughing, shortness of breath, and delayed lung edema; causes chemical burns to the respiratory tract	None known	70 mg/m^3	1 mg/m^3	1 mg/m^3
Piperonal	Dust may cause mechanical irritation; causes skin irritation; may cause irritation of the digestive tract; may cause central nervous system depression, characterized by excitement, followed by headache, dizziness, drowsiness, and nausea — advanced stages may cause collapse, unconsciousness, coma, and possible death due to respiratory failure; inhalation of dust may cause respiratory tract irritation	Central nervous system	None listed	None listed	None listed

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Piperidine	Contact with liquid or vapor causes severe burns and possible irreversible eye damage; contact may cause ulceration of the conjunctiva and cornea; eye damage may be delayed; may cause conjunctivitis; may cause blindness; harmful if absorbed through the skin; may be absorbed through the skin, and if absorbed, causes symptoms similar to those of ingestion; penetration may continue for several days; causes severe skin irritation and burns; harmful if swallowed; may cause severe and permanent damage to the digestive tract; causes gastrointestinal tract burns; can cause nervous system damage; may cause tremors and convulsions; may cause severe irritation of the respiratory tract, with sore throat, coughing, shortness of breath, and delayed lung edema; causes chemical burns to the respiratory tract; may cause effects similar to those described for ingestion; damage may be delayed; may cause bronchial pneumonia	Nervous system			
Platinum	Irritates skin, respiratory system; dermatitis	Eyes, skin, respiratory system	ND	1 mg/m^3	ND
Platinum chloride	Exposure can cause severe allergies affecting the nose, skin, and lungs; irritation and even ulcers can develop in the nose; inhalation may cause platinosis, with symptoms including wheezing, coughing, tightness of the chest, shortness of breath, cyanosis, and pronounced asthmatic symptoms; may cause vomiting and bloody diarrhea; skin contact may cause platinosis, with symptoms including severe irritation, eczema, urticaria, itching, and dermatitis; may cause skin allergy; eye contact may cause irritation, itching, and conjunctival vasodilation	None	None listed	None listed	None listed
Platinum oxide	Causes eye irritation; may cause chemical conjunctivitis; causes skin irritation; may cause gastrointestinal irritation with nausea, vomiting, and diarrhea; causes respiratory tract irritation; can produce delayed pulmonary edema; toxicological properties of this substance have not been fully investigated	None	None listed	None listed	None listed

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Chemical	Effects	Target Organs	IDLH (ppm)	NIOSH REL (ppm)	OSHA PEL (ppm)
Potassium carbonate	Causes irritation to the respiratory tract with symptoms including coughing, shortness of breath; causes irritation to the gastrointestinal tract with symptoms including nausea, vomiting, and diarrhea; may have moderate toxic effects if consumed in large enough quantities; ingestion of large amounts may be corrosive to mouth, throat, and GI tract and may produce abdominal pains, vomiting, diarrhea, and circulatory collapse; contact with dry material causes irritation; in aqueous solution, it is a strong caustic and, as such, may have corrosive effects on the skin; causes extreme irritation, redness, pain, and possibly corneal damage	Eyes, skin, mucous membranes	None listed	None listed	None listed
Potassium cyanide	Contact with eyes may cause severe irritation, and possible eye burns; may be absorbed through the skin in harmful amounts; contact with skin causes irritation and possible burns, especially if the skin is wet or moist; if absorbed, causes symptoms similar to those of ingestion — skin absorption may cause unconsciousness, absorption into the body may cause cyanosis (bluish discoloration of skin due to deficient oxygenation of the blood); may be fatal if swallowed; causes gastrointestinal tract burns; may cause tissue anoxia, characterized by weakness, headache, dizziness, confusion, cyanosis (bluish skin due to deficient oxygenation of the blood), weak and irregular heartbeat, collapse, unconsciousness, convulsions, coma, and death; contains cyanide; human fatalities have been reported from acute poisoning; large doses of cyanide may result in sudden loss of consciousness and prompt death; small doses will prolong the above symptoms 1 to 2 h; can cause central nervous system damage and death, which can be caused by inhalation of high concentrations that may cause central nervous system effects characterized by nausea, headache, dizziness, unconsciousness, and coma; causes respiratory tract irritation; may cause effects similar to those described for ingestion; inhalation may result in symptoms similar to cyanide poisoning that include tachypnea, hyperpnea (abnormally rapid or deep breathing), and dyspnea (labored breathing) followed rapidly by respiratory depression, and pulmonary edema may occur	Central nervous system, respiratory system, cardiovascular system	25 mg/m^3	5 mg/m^3	5 mg/m^3

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Potassium hydroxide	Severe irritant; effects from inhalation of dust or mist vary from mild irritation to serious damage of the upper respiratory tract, depending on the severity of exposure; symptoms may include coughing, sneezing, damage to the nasal or respiratory tract; high concentrations can cause lung damage; Toxic! Estimated lethal dose: 5 g; swallowing may cause severe burns of mouth, throat, and stomach; other symptoms may include vomiting and diarrhea; severe scarring of tissue and death may result; Corrosive! Contact with skin can cause irritation or severe burns and scarring with greater exposures; Highly Corrosive! Causes irritation of eyes with tearing, redness, swelling; greater exposures cause severe burns with possible blindness resulting	Eyes, skin, respiratory system	ND	2 mg/m^3	ND
Pumice	NA	NA	NA	NA	NA
Pyridine	Irritation of eyes; headache, anxiety, dizziness, insomnia; nausea, anorexia; dermatitis; liver, kidney damage	Eyes, skin, central nervous system, liver, kidneys, gastrointestinal tract	1000	5	5
Raney nickel	NA	NA	NA	NA	NA
Sodium	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract; symptoms may include irritation of the nose and throat and labored breathing; may cause lung edema, a medical emergency; is an extremely dangerous, corrosive material that will react immediately with saliva to cause serious burns and possible local combustion and even explosion of hydrogen in the mouth or esophagus; the metal's low melting point can cause further complications; as a corrosive material, can cause serious burns due to almost immediate reaction with water, especially on moist skin; if metal ignites, very deep burns and tissue destruction can occur; corrosive to eyes and may cause redness, pain, blurred vision, and damage from severe alkali burns	None listed	None listed	None listed	None listed
Sodium acetate	May cause irritation to the respiratory tract with symptoms including coughing, sore throat, labored breathing, and chest pain; large doses may produce abdominal pain, nausea, and vomiting; contact may cause irritation, redness, and pain	None	None listed	None listed	None listed

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Chemical	Effects	Target Organs	IDLH (ppm)	NIOSH REL (ppm)	OSHA PEL (ppm)
Sodium bisulfate	Causes eye burns; when substance becomes wet or comes in contact with moisture of the mucous membranes, it will cause irritation; may cause chemical conjunctivitis and corneal damage; causes skin burns; contact with skin causes irritation and possible burns, especially if the skin is wet or moist; may cause skin rash (in milder cases), and cold and clammy skin with cyanosis or pale color; may cause severe and permanent damage to the digestive tract; causes gastrointestinal tract burns; may cause perforation of the digestive tract; may cause systemic effects; may cause severe irritation of the respiratory tract with sore throat, coughing, shortness of breath, and delayed lung edema; causes chemical burns to the respiratory tract; aspiration may lead to pulmonary edema; may cause systemic effects	None	None listed	None listed	None listed
Sodium hydroxide	Causes eye burns; may cause chemical conjunctivitis and corneal damage; causes skin burns; may cause deep, penetrating ulcers of the skin; may cause skin rash (in milder cases), and cold and clammy skin with cyanosis or pale color; may cause severe and permanent damage to the digestive tract; causes gastrointestinal tract burns; may cause perforation of the digestive tract; causes severe pain, nausea, vomiting, diarrhea, and shock; may cause systemic effects; irritation may lead to chemical pneumonitis and pulmonary edema; causes severe irritation of upper respiratory tract with coughing, burns, breathing difficulty, and possible coma; causes chemical burns to the respiratory tract; aspiration may lead to pulmonary edema; may cause systemic effects	Eyes, skin, mucous membranes	10 mg/m^3	2 mg/m^3	2 mg/m^3
Sodium sulfate	May cause eye irritation; may cause skin irritation; ingestion of large amounts may cause gastrointestinal irritation; low hazard for usual industrial handling	None	None listed	None listed	None listed

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Sulfuric acid	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract; symptoms may include irritation of the nose and throat, and labored breathing; may cause lung edema, a medical emergency; corrosive; swallowing can cause severe burns of the mouth, throat, and stomach, leading to death; can cause sore throat, vomiting, diarrhea; circulatory collapse with clammy skin, weak and rapid pulse, shallow respirations, and scanty urine may follow ingestion or skin contact; circulatory shock is often the immediate cause of death; corrosive with dermal contact, showing symptoms of redness, pain, and severe burn; corrosive with eye contact, causing blurred vision, redness, pain, and severe tissue burns, and can cause blindness	Eyes, skin, respiratory system, teeth	15	1	1
Thionyl chloride	Corrosive upon dermal contact, where it is extremely destructive to tissues of the mucous membranes and upper respiratory tract, with symptoms that may include burning sensation, coughing, wheezing, laryngitis, shortness of breath, headache, nausea, and vomiting; inhalation may be fatal as a result of spasm inflammation and edema of the larynx and bronchi, chemical pneumonitis, and pulmonary edema; corrosive upon inhalation, which may cause burning pain in throat, abdominal pain, nausea, and vomiting; corrosive with skin — liquid contact may cause blistering burns, irritation, and pain; vapors may be severely irritating to the skin; Corrosive! Vapors are irritating and may cause damage to the eyes; contact may cause severe burns and permanent eye damage	None known	None listed	1	None listed
Thorium nitrate	Thorium nitrate is a radioactive material; may cause irritation and possible eye damage with eye contact; with skin contact, irritation and dermatitis in sensitive persons is seen; effects of ingestion and inhalation are not known	None listed	None listed	None listed	None listed

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Appendix G: Optical Properties of Inorganic Compounds

Compound	Crystal Form	*n*
Aluminum powder	NA	NA
Aluminum chloride ($AlCl_3 * 6H_2O$)	Col, rhomb, del	1.6
Aluminum oxide (Al_2O_3)	Col, hex	1.768, 1.760
Ammonium chloride (NH_4Cl)	Col, cube	1.642
Ammonium nitrate (NH_4NO_3)	Col, rho, (monocl >32.1°)	
Antimony sulfide (Sb_2S_3)	Blk, rho	3.194, 4.064, 4.303
Barium carbonate ($BaCO_3$)	Wht, rho	1.529, 1.676, 1.677
Barium chlorate [$Ba(ClO_3)_2$]	Col, monocl	1.562, 1.577, 1.635
Barium nitrate [$Ba(NO_3)_2$]	Col, cub	1.572
Barium perchlorate [$Ba(ClO_4)_2$]	Col, hex	1.533
Barium sulfate ($BaSO_4$)	Wht, rho, (monocl)	1.637, 1.638, 1.649
Calcium carbonate ($CaCO_3 * 6H_2O$)	Col, monocl	1.460, 1.535, 1.545
Chromium trioxide (CrO_3)	Rd, rho, del	
Copper sulfate ($CuSO_4 * 5H_2O$)	Blu, tricl	1.514, 1.537, 1.543
Magnesium powder	NA	NA
Magnesium sulfate ($MgSO_4$) anhydrous	Col, rho, cr	1.560
Magnesium sulfate ($MgSO_4 * 7H_2O$) (Epsom salts)	Col, rho or monocl	1.433, 1.455, 1.461
Manganous chloride ($MnCl_2 * 4H_2O$)	Rose, monocl, del	
Manganous carbonate ($MnCO_3$)	Lt brn, rho	
Mercuric chloride ($HgCl_2$)	Col, rho, or wht pdr	1.859
Palladium chloride ($PdCl_2$)	Rd, cu need, del	
Palladium sulfate ($PdSO_4 * 2H_2O$)	Rd-brn, cr, del	
Phosphorus oxychloride ($POCl_3$) or ($P_2O_3Cl_4$)	Col, fum liq	
Phosphorus pentoxide (P_2O_5)	Wht, monocl or pdr, del	
Potassium bicarbonate ($KHCO_3$)	Col, monocl	1.482
Potassium carbonate (K_2CO_3)	Col, monocl, hygro	1.531

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Compound	Crystal Form	*n*
Potassium chlorate ($KClO_3$)	Col, monocl	1.409, 1.517, 1.524
Potassium chromate (K_2Cr0_4)	Hel, rho	1.74
Potassium cyanide (KCN)	Col, cub, wh gran	1.410
Potassium dichromate ($K_2Cr_20_7$)	Red, monocl or tri	1.738
Potassium iodide (KI)	Col or wht, cub	1.677
Potassium nitrate (KNO_3)	Col, rho or trig	1.335, 1.505, 1.506
Potassium perchlorate ($KClO_4$)	Col, rho	1.471, 1.472, 1.476
Sodium acetate ($NaC_2H_3O_2$)	Wht, monocl	1.464
Sodium bicarbonate ($NaHCO_3$)	Wht, monocl, pr	1.526
Sodium borohydrate ($NaBH_4$)	Wht, cub	1.524
Sodium carbonate (Na_2CO_3)	Wht, monocl	1.405, 1.425, 1.440
Sodium chloride (NaCl)	Wht, cub	1.544
Sodium hydride (NaH)	Silver, need	1.470
Sodium nitrate ($NaNO_3$)	Col, trig or rho, bdr	1.587, 1.336
Sodium sulfate ($NaSO_4$)	Monocl	1.480
Strontium nitrate [$Sr(NO_3)_2$]	Col, cub	
Sulfur (S)	Yel, rho	1.957
Thorium nitrate [$Th(NO_3)_4$]	Plates, del	
Zinc powder	NA	NA
Zinc chloride ($ZnCl_2$)	Wht, hex, del	1.681, 1.713

Abbreviations

Brn	Brown	Col	Colorless	Cub	Cubic
Del	Deliquescent	Hex	Hexagonal	Hygro	Hygroscopic
Monocl	Monoclinic	Need	Needles	Ppl	Purple
Pdr	Powder	Rd	Red	Rho	Rhombic
Tetr	Tetragonal	Trig	Trigonal	Wht	White
Cr	Crystals	Pr	Prisms	Fum	Fuming

Source: From *CRC Handbook of Chemistry and Physics*, 68^{th} ed., CRC Press, Boca Raton, FL.

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Appendix H: Crystal Test Reagents

Isomer Crystal Reagents

Reagent	Composition/Preparation
Gold bromide (aqueous)	Dissolve 1g of gold chloride ($HAuCl_4$ * 3 H_2O, i.e., chloroauric acid) and 1 g of sodium bromide in 20 ml of deionized water
Gold bromide (in H_2SO_4)	Combine 1 g of gold chloride with 1.5 ml of 40% HBr; add 28.5 ml of a 2:3 solution of concentrated sulfuric acid and water
Gold chloride (aqueous)	5% (w/v) solution of gold chloride in water
Gold chloride (in H_3PO_4)	5% (w/v) solution of gold chloride in a 1:3 solution of concentrated phosphoric acid and water
Platinum bromide (aqueous)	Dissolve 1 g platinic chloride (H_2PtCl_6 * 6 H_2O, i.e., chloroplatinic acid) with 1 g of sodium bromide in 20 ml deionized water
Platinum bromide (in H_3PO_4)	Combine 1 g of platinic chloride with 1.7 ml of 40% HBr; add 18.3 ml of a 1:3 solution of concentrated phosphoric acid and water
Platinum chloride (aqueous)	5% (w/v) solution of platinic chloride in water
Platinum chloride (in H_3PO_4)	5% (w/v) solution of platinic chloride in a 1:3 solution of concentrated phosphoric acid and water

Inorganic Crystal Reagents

Reagent	Composition/Preparation
Ammonium molybdate	Saturated solution of ammonium molybdate [$(NH_4)_6Mo_7O_{24}$ * $4H_2O$] in concentrated nitric acid
Cropen	Solution A: 5 g of zinc sulfate, 4 g of potassium nitrate dissolved in 20 ml of deionized water Solution B: 0.015% methylene blue in deionized water
Nitron	Dissolved 1 g of nitron [1,2 dihydro 1,4 dipheny 3,5 phenylimino 1,2,3 triazol] in 20 ml of formic acid

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Inorganic Crystal Reagents

Reagent	Composition/Preparation
Platinum chloride	5% (w/v) aqueous solution of platinic chloride (chloroplatinic acid, H_2PtCL_6 * 6 H_2O)
Squaric acid	Saturated aqueous solution of squaric acid (1,2 dihydroxycyclobutenedione)
Strychnine sulfate	Saturated aqueous solution of strychnine sulfate
Uranyl acetate	Solution A (Best for Na^+ and K^+): saturated solution of glacial acetic acid containing 50/50 mixture of uranyl acetate [$UO_2(C_2H_3O_2)$ * 2 H_2O] and zinc acetate [$Zn(C_2H_3O_2)$ * 2 H_2O] Solution B (best for NH_4^+): saturated solution of glacial acetic acid containing uranyl acetate [$UO_2(C_2H_3O_2)$ * 2 H_2O]

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Appendix I: Anion IR Absorbance Table

Anion	Absorbance (cm^{-1})
BO_2^-	1300–1360
B_4O_7	990–1000, 1340–1375
CO_3	1425–1455
HCO_3^-	690–710, 830–840, 990–1010
SCN^-	2020–2100
SiO_3^{-2}	950–1010
NO_2^-	1225–1250
NO_3^-	1340–1390
NH_4^+	1390–1440
PO_4^{-3}	1000–1040
HPO_4^{-2}	1010–1060, 840–900
$H_2PO_4^-$	1025–1090, 900–950
SO_3^{-2}	920–980
SO_4^{-2}	1075–1130
HSO_4^-	845–855, 1030–1070, 1160–1175
$S_2O_3^{-2}$	950–1000, 1100–1120
$S_2O_5^{-2}$	975–990, 1175–1190
$S_2O_8^{-2}$	700–710, 1050–1075, 1275–1300
ClO_3^-	940–970
ClO_4^-	1050–1120
BrO_3^-	795–805
IO_3^-	725–750
$Cr_2O_7^{-2}$	795–860, 860–875
$Cr_2O_7^{-2}$	725–775, 845–890
WO_4^{-2}	790–825
MnO_4^-	890–915
CN^-	2077–2100
OCN^-	2100–2180

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Appendix J: Color Test Reagents

Reagent	Composition/Preparation
Acetic acid (20% aqueous)	To 20 ml acetic acid, add enough water to make 100 ml solution
Acetic acid (67% aqueous)	To 67 ml acetic acid, add enough water to make 100 ml solution
Ammoniated acetone (1.5% solution)	To 1.5 ml concentrated ammonium hydroxide add enough acetone to make 100 ml solution
Ammoniated methanol (1.5% solution)	To 1.5 ml concentrated ammonium hydroxide add enough methanol to make 100 ml solution
Aniline sulfate	Dissolve 0.1 g aniline sulfate in 100 ml concentrated sulfuric acid
Aniline sulfate (aqueous)	Dissolve 5.0 g aniline sulfate in 100 ml deionized water
Barium chloride	Dissolve 5.0 g barium chloride in 100 ml deionized water (5% solution)
Benedict's	Dissolve 1.73 g copper sulfate in 10 ml deionized water; dissolve 17.3 g sodium citrate and 10 g anhydrous sodium carbonate in 60 ml deionized water; filter the citrate solution; add the copper sulfate solution slowly to the citrate solution; dilute the mixture to 100 ml with deionized water
Bismuth iodide (IN H~SO_1)	Prepare a solution of 12.5 ml deionized water and 2.5 ml concentrated sulfuric acid; mix the following ingredients together the following until dissolved: 3.75 g potassium iodide, 1.24 g bismuth nitrate, 0.40 g sodium hypophosphite; refrigerate — over a period of time, the orange-red solution will darken as the iodide decomposes to iodine, at which point the reagent should be discarded
Brucine sulfate	Dissolve 5.0 g brucine sulfate in 100 ml concentrated sulfuric acid
n-Butanol/acetic acid/water	Mix two parts *n*-butanol with one part acetic acid and one part deionized water; prepare fresh before use

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Reagent	Composition/Preparation
Chen's	Reagent 1: dissolve 1.0 g of copper sulfate in a solution of 1 ml acetic acid and 100 ml of deionized water; Reagent 2: 2N solution of sodium hydroxide
Cobalt thiocyanate	Method 1: dissolve 2.0 g of cobalt thiocyanate in 100 ml of deionized water; Method 2: dissolve 2.0 g of cobalt thiocyanate in a solution of 100 ml glycerine and 100 ml of deionized water
Davis	Add 15 ml ethylenediamine to 100 ml 10% aqueous silver nitrate solution
Dilli-Koppanyi	Reagent 1: dissolve 0.1 g of cobaltous acetate tetrahydrate in a solution of 100 ml of methanol and 0.2 ml of glacial acetic acid; Reagent 2: mix 5.0 ml of isopropyl amine with 95 ml methanol
m-Dinitrobenzene	2% solution of *m*-dinitrobenzene in reagent alcohol
Diphenylamine	Dissolve 0.68 g diphenylamine in 45 ml concentrated sulfuric acid; place in ice bath and cautiously add 22.5 ml glacial acetic acid
Dragendorff spray	Mix 2.0 g bismuth subnitrate in a solution of 25 ml acetic acid, and 100 ml dionized water; dissolve 40 g potassium iodide in 100 ml dionized water; mix 10 ml of each of solution with 20 ml acetic acid and 100 ml dionized water
Duquenois	Dissolve 5 drops acetaldehyde and 0.4 g vanillin in 20 ml of 95% ethanol
Ehrlich's	Dissolve 5.0 g of *p*-dimethylaminobenzaldehyde in a solution of 50 ml ethanol and 50 ml of concentrated hydrochloric acid
Ethanol/heptane (95:5)	Mix 95 parts absolute ethanol with 5 parts heptane
Ethanol/hexane (9:1)	Mix 9 parts absolute ethanol with 1 part hexane
Ethylenediamine (15%)	Mix 15 ml of ethylenediamine in 85 ml of deionized water
Fehling's	Dissolve 7.5 g copper sulfate in 100 ml deionized water; dissolve 35 g sodium tartrate and 25 g potassium hydroxide in 100 ml deionized water; mix equal volumes of each solution
Ferric chloride	Mix 1 g ferric chloride in 10 ml deionized water
Fiegel's (sodium nitroprusside)	Dissolve 1.0 g sodium nitroprusside (sodium nitroferricyanide) in 60 ml deionized water; add 10 ml acetaldehyde; dilute solution to 100 ml with deionized water
Froehde's	Dissolve 100 mg of sodium molybdate in 20 ml concentrated sulfuric acid
Hydrochloric acid (0.1 N)	1 ml concentrated hydrochloric acid diluted to 120 ml with deionized water
Hydrochloric acid (3.0 N)	125 ml concentrated hydrochloric acid diluted to 500 ml with deionized water
Hydrochloric acid (15%)	Mix 7.9 ml concentrated HCl into 12.1 ml deionized water
Iodoplatinate spray	10 ml of 10% aqueous solution of chloroplatinic acid; 250 ml of 4% aqueous potassium iodide; combine solutions and add 500 ml deionized water; add 0.75 ml concentrated hydrochloric acid

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Reagent	Composition/Preparation
Le Rosen	Mix 75 ml of concentrated sulfuric acid in 1.5 ml 37% formaldehyde
Liebermann's	Dissolve 10 g potassium nitrite in 100 ml of concentrated sulfuric acid
Mandelins	Dissolve 1.0 g ammonium metal in 100 ml concentrated sulfuric acid
Marquis' (premixed)	Add 8 to 10 drops of 40% formaldehyde solution for each 10 ml concentrated sulfuric acid used
Mecke's	Dissolve 0.25 g selenious acid [H_2SeO)] in 25 ml concentrated sulfuric acid
Mercuric chloride	Dissolve 1.25 g mercuric chloride in 25 ml deionized water
Mercuric iodide	Dilute 27 ml of conconcentrated hydrochloric acid to100 ml with deionized water; add enough mercuric iodide to saturate the acid water solution
Molish solution (*a*-Naphthol)	Dissolve 1.25 g of *a*-Naphthol in reagent alcohol; refrigerate
Nessler	Dissolve 20 g potassium hydroxide in 50 ml deionized water; dissolve 10 g mercuric iodide and 5 g of potassium iodide into 50 ml deionized water; combine solutions
Nitron	Dissolve 3.75 g nitron (diphenylenedianilhydrotriazole) in 75 ml of 88% formic acid
p-DMBA	Dissolve 1.25 g *p*-dimethylaminobenzaldehyde in 25 ml concentrated acetic acid
Silver nitrate	Dissolve 3.75 g of silver nitrate in 75 ml of deionized water
Sodium hydroxide (0.5 N)	Dissolve 1.5 g NaOH in 75 ml deionized water
Sodium hydroxide (2.0 N)	Dissolve 6.0 g NaOH in 75 ml deionized water
Stannous chloride	Dissolve 5.0 g stannous chloride in 10 ml concentrated hydrochloric acid; dilute to 100 ml with deionized water; stannous chloride must be completely dissolved in the HCl before diluting with water
Starch	Saturated solution of hydrolyzed starch in deionized water
Sulfuric acid (75%)	Mix 56.25 ml concentrated sulfuric acid into 18.75 ml deionized water
Sulfuric acid (0.1 N)	Dissolve 1.0 ml concentrated sulfuric acid in 360 ml deionized water
Taratric acid (2.5%)	Make 1.25 g of tartaric acid in 25 ml deionized water
Thymol	Dissolve 0.25 g thymol in 25 ml methanol
Toluene/acetic acid (9:1)	Mix 9 parts toluene with 1 part acetic acid
Triphenyl selenium chloride	Saturated solution in 60 ml deionized water
Triphenyltetrazolium chloride	Dissolve 0.38 g triphenyltetrazolium chloride in 75 ml distilled water
Van Urk's spray	Dissolve 1.0 g *p*-dimethylarninobenzaldehyde in 100 ml of ethanol; add 10 ml concentrated hydrochloric acid
Wagenaar's	Dissolve 1.25 g copper sulfate in 25 ml deionized water; add sufficient ethylenediamine to turn the solution a dark violet color
Wagner's	Dissolve 1.27 g iodine and 2.75 gm potassium iodide in 5.0 ml deionized water; dilute to 100 ml with deionized water

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Reagent	Composition/Preparation
Zwikker's	Reagent 1: dissolve 0.125 g of copper sulfate in 25 ml of deionized water; Reagent 2: mix 2.5 ml of chloroform with 22.5 ml pyridine

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Appendix K: Mass Spec Data of Reaction Mixture Components

	PK1	PK2	PK3	PK4	PK5	MW	Synthesis Route
e-1,5-Diphenyl-2-methyl-1-pentene-4-one	43	91	129	250	57	250	P1 P3
z-1,5-Diphenyl-2-methyl-1-pentene-4-one	43	250	129	57	56	250	P1 P3
Pyrolidine	43	70	71	42	41	71	PC2
n-Acetylamphetamine	44	86	119	91	65	177	A4
Amphetamine	44	91	43	42	65	135	M5
Methylenedioxyamphetamine (MDA)	44	136	135	77	51	179	MD3
n-Methyl 1,2-(methylenedioxy)-4-(3-amonopropyl) benzene	44	162	65	77	135	193	MD5
Cyclohexanone	55	42	98	69	70	98	All PC meth
1,2-(Methylenedioxy)-4-(2-*n*-methylaminopropyl)-benzene	56	191	135	77	57	191	MD3
Morpholine	57	87	56	86	42	87	PC3
Ephedrine	58	69	79	41	59	165	M2 M3
Methylenedioxymethamphetamine (MDMA)	58	136	135	59	77	193	MD3 MD5
n-Methyl-1-[1-(hydroxy)-2-methoxy)]-4-(2-aminopropyl) benzene	58	51	77	137	94	195	MD5
n-Methyl-1-[1,2-(dimethoxy)-4-(2-aminopropyl) benzene	58	152	51	151	59	209	MD5
1-(3,4-Methylenedioxy)pheny;-2-methoxipropane	59	194	135	136	77	194	MD5

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	PK1	PK2	PK3	PK4	PK5	MW	Synthesis Route
n-Formylamphetamine	72	44	118	91	65	163	A1 M5
n,*n*-Dimethylamphetamine	72	44	42	91	56	163	M1 M5
3,4-(Methylenedioxy)-*n*,*n*-dimethylamphetamine	72	56	44	73	58	207	MD3
Bromobenzene	77	71	50	156	158	156	PC1A, 2A, 3A
Benzene	78	77	50	51	52	77	A5
a-Methylbenzyl alcohol	79	77	43	107	51	122	
Piperidine	84	85	55	57	42	84	P1
n-Formylmethamphetamine	86	58	91	56	65	177	M5
3,4-(Methylenedioxy)-*n*-*n*-methylethylamphetamine	86	58	87	56	44	221	MD3
Phenyl-2-propanone	91	134	92	43	65	134	Numerous A and M
Dibenzyl ketone	91	65	39	119	92	210	Num A, M, and P
N,*n*-Di(*b*-phenylyisopropyl)-amine	91	44	162	119	65	253	A1 M5
di-(*b*-Phenylisopropyl)-methylamine	91	58	176	119	42	267	A1 M5
N,*n*-Di-(*b*-phenylisopropyl)-formamide	91	190	119	72	191	281	A1
n-(*b*-Phenylisopropyl)-benzyl methyl ketimine	91	119	160	41	65	251	A4
1-Oxy-1-phenyl-2-(*b*-phenylisopropylimino)-propane	91	119	43	105	77	265	A4
2,4-Dihydroxy-1,5-diphenyl-4-methyl-1-pentene	91	159	131	65	115	268	A4
2-Phenylmethylaziridine	91	104	132	78	51	133	A2 A3
2-Methyl-3-phenylaziridine	91	132	42	105	92	133	A2 A3
Benzyl methyl ketoxime	91	41	92	65	39	149	A3
Benzyl acetate	91	150	65	59	105	150	P1
z-1,3-Diphenyl-2-methyl-1-pentene-4-one	91	130	115	65	159	250	P1 P3
e-1,3-Diphenyl-2-methyl-1-pentene-4-one	91	131	65	105	159	250	P1 P3
Isosafrole glycol	93	151	65	123	152	196	MD1
Phenol	94	66	65	95	67	94	
Camphor	95	81	41	108	152	152	MD5
1-[1-(2-Thienyl)cyclohexyl]-piperidine	97	165	165	206	84	249	PC1B
1-[1-(2-Thienyl)cyclohexyl]-morpholine	97	165	251	208	123	251	PC3B
1-[3,4-(Methylenedioxy)-phenyl]-2-nitro-1-propene	103	160	207	77	102	207	MD2
Phenyl nitropropene	105	115	91	77	116	163	A2
1-Phenyl-2-propanol	105	106	77	79	91	136	P6
Methyl benzoate	105	136	77	51	117	136	P1

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	PK1	PK2	PK3	PK4	PK5	MW	Synthesis Route
Benzaldehyde	106	105	77	51	63	106	P1 P3
1-Morpholinocyclohexene	108	81	167	109	152	167	PC3
z-1-Phenyl;-2-benzyl-1-propene	115	208	91	193	134	208	P1 P3
1-Phenyl-2-benzyl-2-propene	115	208	91	193	178	208	P1 P3
e-1-Phenyl-2-benzyl-2-propene	117	115	91	208	129	208	P1 P3
Dibenzyl methylamine	120	91	42	77	102	211	A1 M5
1-Cyclohexylpiperidine	125	41	167			167	PC1
1-Phenylcyclopentene	129	44	43	128	115	129	PC1
1-Phenylcyclohexene	129	158	115	130	142	158	PC1
e-1,3-Diphenyl-2-methyl-2-pentene-4-one	131	103	77	91	65	250	P1 P3
n-(*b*-Phenylisopropyl)-benzaldimine	132	105	91	77	65	223	A4
1-Phenylcyclohexanol	133	105	176	55	120	176	PC1
a-Benzyl-*n*-methylphenethylamine	134	91	42	119	65	225	M5
z-Phenyl-2-propanone enol acetate	134	43	91	119	105	176	P1
e-Phenyl-2-propanone enol acetate	134	91	43	119	105	176	P1
4-Methyl-1,2-(methylenedioxy)-benzene	135	136	77	79	51	136	MD3
1,2-(Methylenedioxy)-4-propylbenzene	135	77	164	51	79	164	MD1 MD3
3,4-(Methylenedioxy)-benzyl-*n*-methylamine	135	42	51	77	136	165	MD3
3,4-(Methylenedioxy)-phenylpropanone	135	77	51	43	178	178	MD1 MD3
1-[3,4-(Methylenedioxy)]-phenyl-2-propanol	135	136	77	51	106	180	MD3 MD5
3,4-(Methylenedioxy)-benzylmethylketoxime	135	178	176	77	136	283	MD2
n-{*b*-[3,4-(Methylenedioxy)]-phenylmethyl}-3,4-(methylenedioxy)-benzaldimine	135	178	176	77	136	283	MD2
n,*n*-di-[3,4-(Methylenedioxy)-phenylmethyl]-amine	135	150	136	77	51	285	MD2
1-[3,4(Methylenedioxy)]-4-(2-bromopropyl)-benzene	135	77	242	244	51	242	MD5
2-Thienylcyclohexene	135	164	165	136	122	164	PC1B, 2B, 3B
1-Pyrrolidinocyclohexane carbonitrile	135	97	110	136	121	178	PC2
2-Methoxy-4-(2-bromopropyl)-phenol	137	244	246	165	135	244	MD5
1,2-Dimethyl-3-phenylaziridine	146	105	42	132	91	147	M2 M3
Hydroxyskatole	147	146	62	63	89	147	MD2
Piperonal	149	150	121	63	65	150	MD2 MD3
1-[3,4(Methylenedioxy)]-4-(3-bromopropyl)-benzene	149	119	91	163	242	242	MD5

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	PK1	PK2	PK3	PK4	PK5	MW	Synthesis Route
1-Piperidinicyclohexyl carbonitrile (pcc)	149	150	191	164	124	192	PC1
1-Pyrrolidinocyclohexene	150	151	122	136	95	151	PC2
1-Piperidinocyclohexene	150	164	165	136	122	165	PC1
1,2-Dimethoxy-4-(2-bromopropyl)-benzene	151	179	107	258	260	258	MD5
1-Morpholinocyclohexane carbonitrile	151	124	81	136	108	194	PC3
3,4-(Methylenedioxy)-phenylmethanol	152	137	93	65	151	152	MD2
1,3-Diphenyl-2-methyl-2-pentene-4-one	159	91	144	160	141	250	P1 P3
Safrole	162	104	131	103	77	162	MD1 MD3 MD5
1,2-(Dimethoxy)-4-propenylbenzene	162	163	178	147	135	178	MD3
n-Formyl-methylenedioxyamphetamine	162	135	72	44	77	207	MD4
Di-[3,4-(methylenedioxy)]-phenylpropanone	163	135	105	133	77	298	MD2
n-{*b*-[3,4-(Methylenedioxy)]-phenylisopropyl}-3,4-(methylenedioxy)-enzylketimine	163	204	135	105	77	339	MD2
Di-[1–3,4-(methylenedioxy)-phenyl-2-propyl]-amine	163	135	206	105	133	341	MD1
Di-[3,4-(methylenedioxy)-phenyl-2-propyl]-methylamine	163	220	135	105	58	355	MD1
Eugenol	164	77	55	103	149	164	MD5
1,2-Dimethoxy-4-(3-bromopropyl)-benzene	165	162	258	260	119	258	MD5
Diphenylmethane	167	168	152	153	91	168	P3
4-Benzylpyrimidine	169	170	91	115	142	170	
4-Methyl-5-phenylpyrimide	170	169	102	115	116	170	A1
n-{*b*-[3,4-(Methylenedioxy)]-phenylisopropyl}-3,4-(methylenedioxy)-benzaldimine	176	149	177	77	135	311	MD2
4-Allyl-1,2-(dimethoxy)-benzene	178	91	107	103	147	178	MD5
1,2-(Dimethoxy)-4-propenylbenzene	178	107	163	91	103	178	MD5
Bibenzyl	179	180	91	165	89	180	P3
1,3,5-Triphenyl-2,4,6-trimethyl benzene	179	91	257	348	178	348	P1 P3
Cis- or *trans*-stilbene	180	179	178	165	89	180	P1 P3
Trimethoxy-4-(2-bromopropyl) benzene	181	209	288	290	148	277	MD5
2-Methyl-2-phenylmethyl-5-phenyl-2,3-dihydropyrid-4-one	186	91	158	143	187	277	A1

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	PK1	PK2	PK3	PK4	PK5	MW	Synthesis Route
1-(1-Phenylcyclohexyl)-pyrrolidine (PCPV)	186	91	70	152	229	229	PC2A
1-[1-(2-Thienyl)-cyclohexyl]-pyrrolidine	192	97	165	70	235	235	PC2B
1-(1-Phenylcyclohexyl)-piperidine (PCP)	200	91	242	243	186	243	PC1A
1-(1-Phenylcyclohexyl)-morpholine	202	91	245	244	117	245	PC3A
4-Allyl-trimethoxybenzene	208	193	161	133	105	208	MD5
1-Benzyl-3-methylnaphthalene	232	217	108	215	202	232	A1
1,3-Dimethyl-2-phenylnaphthalene	232	215	217	108	202	232	A1
4-Methyl-5-phenyl-(2-phenylmethyl)-pyridine	258	259	243	244	260	259	A1
2-Methyl-3-phenyl-6-phenylmethyl-pyridine	258	259	180	244	260	259	A1
2,4-Dimethyl-3,5-diphenylpyridine	259	260	244	115	215	259	A1
2,6-Dimethyl-3,5-diphenylpyridine	259	260	115	244	101	259	A1
2,4-Dimethy;-3-phenyl-6-phenylmethyl-pyridine	272	273	258	55	57	273	A1
2,4-Diphenyl-3,5-dimethylphenol	274	259	101	165	152	274	

Synthesis Route Key

A1	Amphetamine via Leuckart reaction
A2	Amphetamine via benzaldehyde/nitroethane
A3	Amphetamine via phenylacetone/hydroxylamine
A4	Amphetamine via phenylacetone/ammonia
M2 M3	Methamphetamine via ephedrine reduction
M5	Methamphetamine via Leuckart reaction
P1	Phenylacetone via phenylacetic acid/acetic anhydride
P3	Phenylacetone via phenylacetic acid/lead acetate
PC1	Piperidine/cyclohexane intermediate
PC1A	Phenyl addition
PC1B	Thionyl addition
PC2	Pyroldine/cyclohexane intermediate
PC2A	Phenyl addition
PC2B	Thionyl addition
PC3	Morpholine/cyclohexane intermediate
PC3A	Phenyl addition
PC3B	Thionyl addition
PK1	Largest ion
PK2	2nd largest ion
PK3	3rd largest ion

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PK4	4th largest ion
PK5	5th largest ion
MW	Molecular weight
MD2	MDA via piperonal/nitroethane
MD3	MDMA via amination of MD-P-2-P
MD5	MDMA via safrole/HBr reaction

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Appendix L: Extraction Procedures

Extraction Solubility Guidelines

- Basic drugs are soluble in acidic ($pH < 7$) water solutions, but acidic drugs are not.
- Acidic drugs are soluble in basic ($pH > 7$) water solutions, but basic drugs are not.
- Neutral drugs can be soluble in acidic and basic water solutions.
- Freebase, free acid, and neutral drugs are soluble in organic solvents.
- Freebase and free acid drugs are insoluble in water.
- To determine if a drug is acidic or basic, look at the salt form name:
  - Basic drugs have an acid as part of their salt form [e.g., hydrochloride (HCl), sulfate (H_2SO_4), acetate (CH_3COOH), etc.].
  - Acidic drugs have an alkali metal as part of their salt form name [e.g., sodium (Na) or potassium (K)].
  - Neutral drugs do not have an associated salt form.

Particle Picking

- Remove particles that appear to be the substance in question with tweezers.
- Analyze by IR spectroscopy.

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Dry Extraction

- Place powder sample in an organic solvent in which the component of interest is soluble and the diluents and adulterants are not.
- Isolate and evaporate the solvent.
- Analyze by IR spectroscopy.

Dry Wash

- Place powder sample in an organic solvent in which the component of interest is NOT soluble and the diluents and adulterants ARE.
- Remove the solvent from the dry solid.
- Analyze by IR spectroscopy.
- Multiple solvents may be required to remove all diluents and adulterants.

General Liquid/Liquid Extraction Procedure

- Dissolve the sample in an acidic aqueous solution. (The basic drugs will dissolve into the acidic aqueous solution.)
- Add an organic solvent, and agitate. (The acidic and neutral drugs will dissolve into the organic solvent.)
- Allow the liquids to separate.
- Remove and discard the organic solvent.
- Make the aqueous liquid basic. (The basic drugs will come out of solution.)
- Add an organic solvent, and agitate. (The basic drugs will dissolve into the organic solvent.)
- Allow the liquids to separate.
- Remove and retain the organic solvent.
- Analyze the solvent.
  - The organic solvent can be analyzed by GCMS.
  - Or, the solvent can be evaporated, and the residue can be analyzed via IR spectroscopy.

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General Ion-Pairing Extraction Technique

- Dissolve the sample in an acidic aqueous solution with a high concentration of halide ions. (The use of HCl or the addition of a chloride salt will provide the needed environment. The basic drugs will dissolve into the acidic solution.)
- Add a chlorinated solvent (chloroform) and agitate. (The ion-pairing drug will extract into the organic solvent along with the acidic and neutral drugs.)
- Allow the liquids to separate.
- Remove and save the organic solvent.
- Add an acidic aqueous solvent, void of chloride ions, to the organic solution, and agitate. (The ion-pairing drugs will dissolve into the acidic solution.)
- Allow the liquids to separate.
- Remove and discard the organic solvent.
- Make the aqueous liquid basic. (The ion-pairing drugs will not be soluble in the basic aqueous solution.)
- Add an organic solvent, and agitate. (The ion-pairing drugs will dissolve into the organic solvent.)
- Allow the liquids to separate.
- Remove and retain the organic solvent. (The organic solvent can be analyzed by GCMS. The solvent can be evaporated, and IR spectroscopy can be used to analyze the residual freebase drug.)

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Appendix M: General Calculation Equations

Geometric Shape Volumes

Cylinder	= * radius * radius * height
	= 3.1415 * (diameter/2) * (diameter/2) * height
	= 0.78 * diameter * diameter * height
Cone	= 0.33 * radius * radius * height
	= 0.33 * 3.1415 * (diameter/2) * (diameter/2) * height
	= 0.26 * diameter * diameter * height
Sphere	= 1.33 * π * radius * radius * radius
	= 1.33 * 3.1415 * (diameter/2) * (diameter/2) * (diameter/2)
	= 0.522 * diameter * diameter * diameter

Gravimetric Quantitation

Solid Samples

% Compound	= (Weight extracted compound/Original sample weight) * 100
Amount of compound in original container	= % Compound * Original bulk weight

Liquid Samples

Sample concentration	= Weight of extracted compound/Volume of extracted sample
Amount of compound in original container	= Sample concentration * Original volume of seized item

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Serial Dilution Quantitation

Line equation $Y = (m*x) + b$

Line slope (m) = (Standard $\text{concentration}_{\text{max.}}$ – Standard $\text{concentration}_{\text{min.}}$)/(Standard instrument $\text{response}_{\text{max.}}$ – Standard instrument $\text{response}_{\text{min.}}$)

Concentration (y) = [Line slope (m) * Instrument response (x)] + Y intercept (b)
= {[Standard $\text{concentration}_{\text{max.}}$ – Standard $\text{concentration}_{\text{min.}}$/(Standard instrument $\text{response}_{\text{max.}}$ – Standard instrument $\text{response}_{\text{min.}}$)] * Unknown instrument response} + 0 (assumed)

Percentage = (Calculated concentration/Sample concentration) * 100
= [Calculated concentration/(Sample weight/Sample volume)] * 100

Single Standard Solution Quantitation

Concentration unknown = (Peak area unknown * concentration standard)/Peak area standard

Concentration unknown = (Peak area unknown * Peak area internal standard of standard * concentration standard)/(Peak area standard * Peak area internal standard of unknown)

Production Estimates

Conversion factor (n) = Molecular weight final product/molecular weight precursor chemical

Weight theoretical = n * Weight precursor chemical

Weight actual = n * Weight precursor chemical * percentage yield estimated

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Appendix N: Conversion Factor Table

	P2P	Amphetamine HCl	Methamphetamine HCl	MDA HCl	MDMA HCl	PCP
Acetaldehyde			4.20			
Acetic acid	2.23	3.06	3.08			
Acetonotrile		4.48				
Allyl benzene		1.55				
Allyl chloride		2.42				
Ammonia		10.2		12.6		
Ammonium formate		2.92		3.41		
Benzaldehyde	1.26	1.73	1.74			
Benzene	1.52	2.37	2.38			
Benzyl chloride		1.46				
Benzyl cyanide	1.14	1.57	1.56			
Bromobenzene						2.31
Chloroacetone	1.45	2.00	2.01			
Cyclohexanone						2.33
Ephedrine HCl			0.92			
Ethyl acetate	1.52	2.09	2.10			
Formamide		4.08		4.77		
Hydroxylamine HCl		2.66				
Isosafrole				1.32	1.41	
Methylamine			5.96		7.38	
Methylene chloride				2.52	2.69	
3,4-Methylenedioxy-phenyl-2-propanone				1.20	1.53	
n-Methylformamide			3.13		3.88	
Nitroethane	1.78	2.45	2.46			
Phenylpropanolamine		1.21				
Phenyl-2-propanol	0.99	1.36	1.37			

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	P2P	Amphetamine HCl	Methamphetamine HCl	MDA HCl	MDMA HCl	PCP
Phenyl-2-propanone		1.37	1.38			
Phenylacetic acid	0.98	1.35	1.36			
Piperidine						2.46
Piperonal				1.43		
Safrole				1.32	1.41	
a-Acetylphenyl-acetonitrile	0.84	1.16	1.17			

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Appendix O: List of Explosive Materials

The following notice was copied from an information bulletin located on the Internet. It provides a list of the materials that are considered to be explosive by the Bureau of Alcohol, Tobacco, and Firearms.

Department of the Treasury
Bureau of Alcohol, Tobacco, and Firearms
[Notice No.]
Commerce in Explosives

Pursuant to the provisions of Section 841(d) of Title 18, United States Code, and 27 CFR 55.23, the Director, Bureau of Alcohol, Tobacco, and Firearms, must publish and revise at least annually in the *Federal Register* a list of explosives determined to be within the coverage of 18 U.S.C. Chapter 40, Importation, Manufacture, Distribution and Storage of Explosive Materials. This Chapter covers not only explosives, but also blasting agents and detonators, all of which are defined as explosive materials in section 841(c) of Title 18, United States Code. Accordingly, the following is the 1989 List of Explosive Materials subject to regulation under 18 U.S.C. Chapter 40, which includes both the list of explosives (including detonators) required to be published in the *Federal Register* and blasting agents. The list is intended to also include any and all mixtures containing any of the materials in the list. Materials constituting blasting agents are marked by an asterisk. While the list is comprehensive, it is not all inclusive. The fact that an explosive material may not be on the list does not mean that it is not within the coverage of the law if it otherwise meets the statutory definitions in Section 841 of Title 18, United States Code. Explosive materials are listed alphabetically by their

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common names followed by chemical names and synonyms in brackets. This revised list supersedes the List of Explosive Materials dated December 29, 1988 (53 FR 52561) and will be effective as of (the date of publication in the *Federal Register*).

List of Explosive Materials

A

Acetylides of heavy metals
Aluminum containing polymeric propellant
Aluminum ophorite explosive
Amatex
Amatol
Ammonal
Ammonium nitrate explosive mixtures (cap sensitive)
*Ammonium nitrate explosive mixtures (non cap sensitive)
Aromatic nitro-compound explosive mixtures
Ammonium perchlorate having particle size less than 15 microns
Ammonium perchlorate composite propellant
Ammonium picrate [picrate of ammonia, Explosive D]
Ammonium salt lattice with isomorphously substituted inorganic salts
*ANFO [ammonium nitrate-fuel oil]

B

Baratol
Baronol
BEAF [1, 2-bis (2, 2-difluoro-2- nitroacetoxyethane)]
Black powder
Black powder based explosive mixtures
*Blasting agents, nitro-carbo-nitrates, including non cap sensitive slurry and water-gel explosives
Blasting caps
Blasting gelatin
Blasting powder
BTNEC [bis (trinitroethyl) carbonate]
BTNEN [bis (trinitroethyl) nitramine]
BTTN [1,2,4 butanetriol trinitrate]
Butyl tetryl

C

Calcium nitrate explosive mixture

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Cellulose hexanitrate explosive mixture
Chlorate explosive mixtures
Composition A and variations
Composition B and variations
Composition C and variations
Copper acetylide
Cyanuric triazide
Cyclotrimethylenetrinitramine [RDX]
Cyclotetramethylenetetranitramine [HMX]
Cyclonite [RDX]
Cyclotol

D

DATB [diaminotrinitrobenzene]
DDNP [diazodinitrophenol]
DEGDN [diethyleneglycol dinitrate]
Detonating cord
Detonators
Dimethylol dimethyl methane dinitrate composition
Dinitroethyleneurea
Dinitroglycerine [glycerol dinitrate]
Dinitrophenol
Dinitrophenolates
Dinitrophenyl hydrazine
Dinitroresorcinol
Dinitrotoluene-sodium nitrate explosive mixtures
DIPAM
Dipicryl sulfone
Dipicrylamine
DNDP [dinitropentano nitrile]
DNPA [2,2-dinitropropyl acrylate]
Dynamite

E

EDDN [ethylene diamine dinitrate]
EDNA
Ednatol
EDNP [ethyl 4,4-dinitropentanoate]
Erythritol tetranitrate explosives
Esters of nitro-substituted alcohols
EGDN [ethylene glycol dinitrate]
Ethyl-tetryl

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Explosive conitrates
Explosive gelatins
Explosive liquids
Explosive mixtures containing oxygen releasing inorganic salts and hydrocarbons
Explosive mixtures containing oxygen releasing inorganic salts and nitro bodies
Explosive mixtures containing oxygen releasing inorganic salts and water insoluble fuels
Explosive mixtures containing oxygen releasing inorganic salts and water soluble fuels
Explosive mixtures containing sensitized nitromethane
Explosive mixtures containing tetranitromethane (nitroform)
Explosive nitro compounds of aromatic hydrocarbons
Explosive organic nitrate mixtures
Explosive powders

F

Fulminate of mercury
Fulminate of silver
Fulminating gold
Fulminating mercury
Fulminating platinum
Fulminating silver

G

Gelatinized nitrocellulose
Gem-dinitro aliphatic explosive mixtures
Guanyl nitrosamino guanyl tetrazene
Guanyl nitrosamino guanylidene hydrazine
Guncotton

H

Heavy metal azides
Hexanite
Hexanitrodiphenylamine
Hexanitrostilbene
Hexogen [RDX]
Hexogene or octogene and a nitrated *N*-methylaniline
Hexolites
HMX [cyclo-1,3,5,7-tetramethylene-2,4,6,8-tetranitramine; Octogen]

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Hydrazinium nitrate/hydrazine/aluminum explosive system
Hydrazoic acid

I

Igniter cord
Igniters
Initiating tube systems

K

KDNBF [potassium dinitrobenzo-furoxane]

L

Lead azide
Lead mannite
Lead mononitroresorcinate
Lead picrate
Lead salts, explosive
Lead styphnate [styphnate of lead, lead trinitroresorcinate]
Liquid nitrated polyol and trimethylolethane
Liquid oxygen explosives

M

Magnesium ophorite explosives
Mannitol hexanitrate
MDNP [methyl 4,4-dinitropentanoate]
MEAN [monoethanolamine nitrate]
Mercuric fulminate
Mercury oxalate
Mercury tartrate
Metriol trinitrate
Minol-2 [40% TNT, 40% ammonium nitrate, 20% aluminum]
MMAN [monoethylamine nitrate]; methylamine nitrate
Mononitrotoluene-nitroglycerin mixture
Monopropellants

N

NIBTN [nitroisobutametriol trinitrate]
Nitrate sensitized with gelled nitroparaffin
Nitrated carbohydrate explosive
Nitrated glucoside explosive

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Nitrated polyhydric alcohol explosives
Nitrates of soda explosive mixtures
Nitric acid and a nitro aromatic compound explosive
Nitric acid and carboxylic fuel explosive
Nitric acid explosive mixtures
Nitro aromatic explosive mixtures
Nitro compounds of furane explosive mixtures
Nitrocellulose explosive
Nitroderivative of urea explosive mixture
Nitrogelatin explosive
Nitrogen trichloride
Nitrogen tri-iodide
Nitroglycerine [NG, RNG, nitro, glyceryl trinitrate, trinitroglycerine]
Nitroglycide
Nitroglycol (ethylene glycol dinitrate, EGDN)
Nitroguanidine explosives
Nitroparaffins Explosive Grade and ammonium nitrate mixtures
Nitronium perchlorate propellant mixtures
Nitrostarch
Nitro-substituted carboxylic acids
Nitrourea

O

Octogen [HMX]
Octol [75% HMX, 25% TNT]
Organic amine nitrates
Organic nitramines

P

PBX [RDX and plasticizer]
Pellet powder
Penthrinite composition
Pentolite
PYX (2,6-bis(picrylamino)-3,5-dinitropyridine
Perchlorate explosive mixtures
Peroxide based explosive mixtures
PETN [nitropentaerythrite, pentaerythrite tetranitrate, pentaerythritol tetranitrate]
Picramic acid and its salts
Picramide
Picrate of potassium explosive mixtures

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Picratol
Picric acid (manufactured as an explosive)
Picryl chloride
Picryl fluoride
PLX [95% nitromethane, 5% ethylenediamine]
Polynitro aliphatic compounds
Polyolpolynitrate-nitrocellulose explosive gels
Potassium chlorate and lead sulfocyanate explosive
Potassium nitrate explosive mixtures
Potassium nitroaminotetrazole

R

RDX [cyclonite, hexogen, T4, cyclo-1,3,5,-trimethylene-2,4,6,-trinitramine; hexahydro-1,3,5-trinitro-*S*-triazine]

S

Safety fuse
Salts of organic amino sulfonic acid explosive mixture
Silver acetylide
Silver azide
Silver fulminate
Silver oxalate explosive mixtures
Silver styphnate
Silver tartrate explosive mixtures
Silver tetrazene
Slurried explosive mixtures of water, inorganic oxidizing salt, gelling agent, fuel and sensitizer (cap sensitive)
Smokeless powder
Sodatol
Sodium amatol
Sodium azide explosive mixture
Sodium dinitro-ortho-cresolate
Sodium nitrate-potassium nitrate explosive mixture
Sodium picramate
Squibs
Styphnic acid explosives

T

Tacot [tetranitro-2,3,5,6-dibenzo-1,3a,4,6a-tetrazapentalene]
TATB [triaminotrinitrobenzene]
TEGDN [triethylene glycol dinitrate]

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Tetrazene [tetracene, tetrazine, l(5-tetrazolyl)-4-guanyl tetrazene hydrate]
Tetranitrocarbazole
Tetryl [2,4,6 tetranitro-*N*-methylaniline]
Tetrytol
Thickened inorganic oxidizer salt slurried explosive mixture
TMETN (trimethylolethane trinitrate)
TNEF [trinitroethyl formal]
TNEOC [trinitroethylorthocarbonate]
TNEOF [trinitroethyl orthoformate]
TNT [trinitrotoluene, trotyl, trilite, triton]
Torpex
Tridite
Trimethylol ethyl methane trinitrate composition
Trimethylolthane trinitrate-nitrocellulose
Trimonite
Trinitroanisole
Trinitrobenzene
Trinitrobenzoic acid
Trinitrocresol
Trinitro-meta-cresol
Trinitronaphthalene
Trinitrophenetol
Trinitrophloroglucinol
Trinitroresorcinol
Tritonal

U

Urea nitrate

W

Water bearing explosives having salts of oxidizing acids and nitrogen bases, sulfates, or sulfamates (cap sensitive)
Water-in-oil emulsion explosive compositions

X

Xanthamonas hydrophilic colloid explosive mixture

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For further information contact:

Firearms and Explosives Operations Branch
Bureau of Alcohol, Tobacco and Firearms
1200 Pennsylvania Avenue, NW
Washington, D.C., 20226
202-789-3027

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Appendix P: Sphere Volume Estimates

Flask Volume [Liquid Height (cm)]	1000 ml	2000 ml	3000 ml	5000 ml	12,000 ml	22,000 ml	50,000 ml	72,000 ml
0.5	5	6	7	9	11	14	18	21
1.0	19	24	28	33	45	55	73	83
1.5	42	53	62	74	100	123	163	185
2.0	79	93	107	129	176	217	288	326
2.5	109	141	164	198	271	336	447	506
3.0	152	199	232	280	385	478	638	724
3.5	200	264	309	348	518	644	862	979
4.0	252	336	394	481	667	832	1117	1270
4.5	308	414	488	597	833	1042	1402	1596
5.0	366	497	589	723	1015	1273	1717	1957
5.5	426	585	696	859	1212	1524	2061	2351
6.0	487	676	808	1002	1422	1794	2433	2778
6.5	549	770	925	153	1646	2082	2832	3238
7.0	609	866	1046	1310	1882	2388	3258	3728
7.5	669	963	1170	1473	2129	2710	3709	4249
8.0	726	1061	296	1641	2388	3048	4185	4799
8.5		1158	1423	1812	2556	3401	4685	5378
9.0		1253	1551	1987	2933	3769	5208	5984
9.5		1347	1678	2165	3218	4150	5753	6618
10.0			1804	2344	3510	4542	6319	7278
10.5			1929	2523	3810	4948	6909	7963
11.0			2050	2703	4115	5364	7513	8673
11.5			2168	2881	4424	5790	8139	9407
12.0			2281	3058	4738	6225	8783	10,163
12.5				3232	5055	6668	9444	10,942
13.0				3402	5357	7120	10,121	10,163
13.5				3569	5696	7577	10,815	12,562

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Flask Volume [Liquid Height (cm)]	1000 ml	2000 ml	3000 ml	5000 ml	12,000 ml	22,000 ml	50,000 ml	72,000 ml
14.0				3731	6017	8041	11,523	13,402
14.5					6340	8510	12,245	14,261
15.0					6661	8984	12,980	15,137
15.5					6980	9860	13,728	16,031
16.0					7297	9940	14,487	16,941
16.5					7610	10,421	15,257	17,867
17.0					7920	10,903	16,036	18,807
17.5					8224	11,358	16,825	19,761
18.0					8522	11,867	17,622	20,729
18.5						12,347	18,426	21,708
19.0						12,825	19,237	22,699
19.5						13,299	20,054	23,700
20.0						13,771	20,875	24,711
20.5						14,236	21,701	25,731
21.0						14,697	22,530	26,758
21.5							23,361	27,794
22.0							24,194	28,835
22.5							25,028	29,882
23.0							25,862	30,934
23.5							26,695	31,990
24.0							27,526	33,049
24.5							28,354	34,110
25.0							29,190	35,173
25.5							30,001	36,236
26.0							30,817	37,299
26.5							31,627	38,361
27.0							32,431	39,421
27.5								40,479
28.0								42,533
28.5								42,583
29.0								43,627
29.5								44,666
30.0								45,967

Source: From *Microgram*, 24, 7, 184, July, 1991.

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Appendix Q: Glossary

Accident	An unforeseen happening resulting in damage to people or property.
Accuracy	The ability of a measurement to match the actual value of the quantity being measured; correctness.
Acid	Compounds that readily donate a proton (hydrogen) to a chemical reaction. Substances with a pH < 7.
Acute effects	Effects of exposure to chemical hazards in high concentrations for a short duration.
Adulterant	A substance used to increase the mass of a controlled substance. These substances produce a physiological effect on the body and are used to give the illusion that there is more controlled substance present than is actually there.
Alkaloid	A class of substances readily formed in the tissues of plants and the bodies of animals, e.g., morphine and codeine are alkaloids of opium.
Anion	A negatively charged group of atoms. e.g., OH^-.
Aqueous	Made from, or by means of, water.
Associative evidence	Evidence that establishes a relationship between two items.
Base	Compounds that readily accept a proton (hydrogen) in a chemical reaction.
Beyond a reasonable doubt	The proof of guilt required for conviction of a criminal defendant. A reasonable doubt exists when a fact finder cannot say with moral certainty that a person is guilty, or a particular fact exists. It must be more than an imaginary doubt, and it is often defined judicially as such doubt that would cause a reasonable person to hesitate before acting in a matter of importance.
Birefringence	The resolution or splitting of a light wave into two unequally reflected or transmitted waves by an optically anisotropic medium such as calcite or quartz. Also called double refraction.

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BLEVE	Boiling Liquid Expanding Vapor Explosion — A type of mechanical explosion in which the pressure from the expanding vapors of a boiling liquid compromise the structural integrity of the container of a closed system.
Boiling plateau	The temperature a mixture of boiling liquids maintains until an individual component has evaporated.
Boiling point	The temperature at which a liquid changes into a vapor.
Burden of proof	The responsibility of proving a disputed charge or allegation.
Carcinogen	A chemical agent capable of causing the development of cancerous cells.
Cation	A positively charged group of atoms. e.g., NH_4^+.
Chronic effects	Effects of exposure to chemical hazards in low concentrations for a long duration or extended period of time.
Class characteristic	A feature of an item that is unique to a group of items.
Combustible liquid	A compound with a flash point above 100°C.
Compatible chemicals	Chemicals that can remain in close or permanent contact without reacting.
Confined space	A space with limited entry or exit openings and unfavorable ventilation that is not intended for continuous occupancy.
Confirmatory test	A documentable examination that provides data considered specific to the compound under examination.
Controlled substance	Any substance, commonly drugs, that when possessed or used is regulated.
Conversion process	Changing a raw material into the finished product by making minor changes in the molecule or its salt form.
Corrosive	Chemicals that can cause visible damage to metals, plastics, or other materials (especially your skin).
Deductive reasoning	Using nonspecific details to infer a specific fact.
Deflagration	An explosion with a reaction rate of less than 1000 m/sec.
Depressant	A drug that reduces excitability and calms a person.
Detonation	An explosion with a reaction rate of greater than 1000 m/sec.
Diluent	An inert substance used to increase the mass of the controlled substance. These substances have no physiological effect on the body and are used to give the illusion that there is more controlled substance present than actually is present.
Distillation	The separation of a liquid from a solid or other liquid using evaporation followed by condensation.
Drug	A substance other than food that is intended to affect the structure or function of the body.
Embryonic toxin	A chemical agent that can cause fetal death.
Explosion	A rapid chemical change that produces a large amount of heat and gas.
Explosive chemicals	Compounds that undergo a rapid chemical change that releases a large amount of heat and gas.
Explosives (27 CFR 55.11)	Any chemical compound, mixture, or device, the primary or common purpose of which is to function by explosion.
Extraction	The act of separating a constituent from the whole.
Extraction process	Removes raw material from a mixture without chemically changing the material being extracted.

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Flammable	A compound with a flash point below 100°F.
Flash point	The lowest temperature at which a source of ignition will ignite the vapors above a flammable liquid.
Fourier transform	A technique for expressing a waveform as a weighted sum of sines and cosines.
Explosive/flammable limits	The atmosphere's fuel and air mixture range that will support combustion. The flammable range refers to an unconfined atmosphere. The explosive range refers to a confined atmosphere.
Fireworks (27 CFR 55.11)	Any composition or device designed to produce a visual or an audible effect by combustion, deflagration, or detonation and that meets the definition of "consumer fireworks" or "display fireworks."
Gas chromatography	The use of gas flowing through a coated tube to separate compounds by their sizes, weights, and chemical reactivities with the column coating.
Gravimetric quantitation	Using the ratio of pre- and postextraction weights to determine concentration.
Hallucinogen	A psychoactive drug that induces hallucinations or alters sensory experiences.
Hydrogenation	A chemical reaction that adds hydrogen to a substance through the direct use of gaseous hydrogen.
IDLH	Immediately Dangerous to Life or Health
Incompatible chemicals	Chemicals that when combined generate heat and cause a fire or explosion, form a toxic gas or vapor, form a substance that is more toxic than the original compounds, disperse a toxic mist or dust, produce a violent chemical reaction, or produce any combination thereof.
Individual characteristic	A feature that is unique to a specific item.
Inductive reasoning	Using specific facts to infer a general conclusion.
Infrared spectroscopy	The use of the absorption of infrared radiation to produce a chemical fingerprint of a substance.
LD50	The concentration at which a substance will be lethal to 50% of the test population. It is usually expressed as weight of substance per weight of test subject (e.g., 5 mg/kg rat).
Lower explosive/flammable limit	The minimum atmospheric concentration of a substance that will explode (confined space) or ignite (unconfined space). Concentrations below this level are said to be fuel poor and will not explode or ignite.
Macroscopic examination	Visual examination, generally performed with the unaided eye, used to identify class characteristics.
Manufacture (21 CFR 1300.01)	"… the producing, preparing, propagating, compounding or processing of a drug or other substance or the packaging or repackaging of such substance or labeling or relabeling of the commercial container of such…"
Manufacturer (27 CFR 55.11)	Any person engaged in the manufacturing of explosive materials for purposes of sale or distribution or for his own use.
Mass spectroscopy	The use of molecular fragment (ion) patterns to produce a chemical fingerprint of a substance.

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Melting point	The temperature at which a solid changes into a liquid.
Microscopic examination	Visual examination, performed utilizing some type of magnification, used to identify individual characteristics.
Mutagen	A chemical agent that can cause mutations at a greater frequency than normally expected. The mutation can be a result of alteration of the genetic code. Many mutagens are carcinogens.
Narcotic	An addictive substance that reduces pain, alters mood and behavior, and usually induces sleep or stupor.
Organic	The class of chemical compounds having a carbon basis; hydrocarbons are organic compounds.
Oxidizer	Compounds that provide oxygen to a reaction.
PEL	Permissible Exposure Limit.
Poison	A substance that in low concentrations will cause death or injury upon ingestion.
Polymorphism	Crystallization of a compound in at least two distinct forms.
Precision	The ability to achieve the same result; reproducibility.
Precursor chemical	A raw material that becomes a part of the finished product.
Preponderance of evidence	The least demanding standard of proof and is used for most civil actions and some criminal defenses (as insanity). Clear and convincing proof is a more demanding standard of proof and is used in certain civil actions (as a civil fraud suit). Proof beyond a reasonable doubt is the most demanding standard and the one that must be met for a criminal conviction.
Pyrophoric	Chemicals that react with the air and may spontaneously ignite.
Pyrotechnic compositions (27 CFR 55.11)	A chemical mixture, that upon burning and without explosion, produces visible brilliant displays, bright lights, or sounds.
Qualitative analysis	Analytical technique used to determine the composition of a substance or mixture.
Quantitative analysis	Analytical technique used to determine the concentration of one or more of the components of a mixture.
Racemic mixture	A combination of the different types of stereoisomers of the same compound.
Reagent chemical	A chemical that reacts with one or more of the precursor chemicals but does not become part of the finished product.
Reducer	A compound that can remove oxygen from or add hydrogen to a compound.
Reflux	A controlled boiling process in which the evaporated liquid is condensed and returned to the reaction mixture.
Relative retention time	The ratio of the retention time of the substance of interest divided by the retention time of an internal standard run on the same instrument at the same time.
Retention time	The time required for a substance to travel from the injection port to the detector.
Screening (preliminary) test	An examination that provides information concerning the class characteristics of the substance under examination.
Solvent	A chemical that is used to dissolve solid precursors or reagents, to dilute reaction mixtures, and to separate or purify other chemicals. They do not react with precursor or reagent chemicals.

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Stereoisomers	Compounds with identical structural formulas with differences that are in the way the molecule is arranged in space.
Stimulant	A drug that produces a temporary increase of the functional activity or efficiency of an organism or any of its parts.
Structural isomers	Compounds that contain the same number and type of atoms but differ in the order in which the atoms are arranged. The types of structural isomers include chain, positional, and functional groups.
Synthesis process	A chemical reaction or series of chemical reactions in which molecules or parts of molecules are combined to create a new molecule.
Tableting process	The act of placing the finished product into dosage forms or into smaller salable units for distribution.
Teratogen	A chemical agent that produces a system malfunction, generally in the form of nonlethal mutations or tumors.
Thin-layer chromatography	The use of a solvent traveling through a porous medium to separate compounds by their chemical reactivity with the solvent.
TLV	Threshold Limit Value — .
Ultraviolet spectroscopy	The use of the absorption of ultraviolet radiation to classify a substance.
Upper explosive/flammable limit	The maximum atmospheric concentration of a substance that will explode (confined space) or ignite (unconfined space). Concentrations above this level are said to be fuel rich and will not explode or ignite.
Vapor density	The ratio of the density of a gas or vapor to the density of ambient air.
Water reactive	Chemicals that hydrolyze with water forming flammable, corrosive, or toxic products.

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Appendix R: Clandestine Drug Lab Reference Material

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